

THE TECHNOLOGY
OF SUGAR

J. G. MINTOSH

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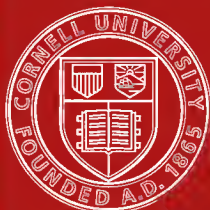
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THE TECHNOLOGY OF SUGAR

THE TECHNOLOGY OF SUGAR

BY

JOHN GEDDES M'INTOSH

LATE LECTURER ON AGRICULTURAL CHEMISTRY
THE POLYTECHNIC, REGENT STREET

WITH EIGHTY-THREE ILLUSTRATIONS
AND SEVENTY-SIX TABLES

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PREFACE

THE Author would have liked to have dealt with the cultivation of the sugar beet, the influence of pedigree seed on the sugar content of the beet, and to have described more fully the culture of the sugar cane; but he deemed it better to devote the bulk of the space at his disposal to the wants of the Sugar-works Chemist, the Engineer, and the Public Analyst. Sugar-cane and sugar-beet cultivation are, in fact, separate industries from sugar manufacture and refining. He trusts that the book may be useful not only to those for whom it is primarily intended, but to all those who have to do with the subject—*e.g.* Excise and Custom Officers, and all intelligent members of society who may wish to know something more about sugar. Now that the Bounty system is about to be abolished, the British sugar manufacturer must look to his laurels. The Bounty system, however prejudicial, was not the only cause of the decline and fall of sugar refining in Britain and sugar manufacture in British Colonies. Obsolete machinery and methods contributed much to the decadence of the industry.

As the book is not intended for the purely theoretical chemist, therefore in their present incomplete state of transition it has not been thought necessary to enter either into such questions as the synthesis of sugars, or to discuss the newer theories and classification of saccharine bodies. As to the chemical nomenclature used, the Author has exercised his right to use what seemed to him best suited for the occasion. But when will theoretical reviewers realise that *nomenclature* is not *chemistry*, and that a man may be a sound chemist whatever system he uses, and that it does not necessarily follow that if he uses one system he is ignorant of the other? The book has not been burdened with voluminous tables of imports of sugar and other statistics of only ephemeral interest, nor has it been

made a record of inventions of machinery and processes. Those interested can get all the abridgments of sugar-patent specifications *ab initio* for a nominal sum, and the statistics in regard to sugar are to be found for the preceding five years in the *Annual Statement of Trade* prepared by H.M. Customs.

The Sugar-works Engineer and Chemist will find it to his advantage to supplement this treatise with Hausbrand's *Evaporating, Condensing, and Cooling Apparatus* (published by the same firm as this treatise), which is a perfect storehouse of information in regard to everything pertaining to evaporation and the thermo-dynamical laws by which it is regulated.

The Author's task has in many instances been limited to assimilating the works of his predecessors—Ure, Wallace, Brande, Scoffern, Newlands, Muspratt, and Gill, in Britain; Horsin-Deon, Sidersky, and Cambier, in France; Wiley and Spencer, in America; Jelinek and others, in Austria; Classen and Hausbrand, in Germany. To each of these now alive, and the other living writers to whom the Author is indebted, he returns his heartfelt thanks; but the special literature of this subject is very voluminous, and much of it does not admit of useful condensation.

We all hope for a speedy revival of the Sugar Industry both at home and in our Colonies; and should this volume assist in some measure, it will not have been written in vain.

J. G. M.

LONDON, *October* 1903.

TABLE OF CONTENTS



	PAGE
PREFACE	v
TABLE OF CONTENTS	vii
LIST OF ILLUSTRATIONS	xiii

SECTION I

CLASSIFICATION OF SUGARS

CHAPTER I

The Chemistry of Sucrose, Lactose, Maltose, Glucose, and Levulose, and their Saccharimetric, Volumetric, and Gravimetric Determination	1
--	---

SECTION II

THE BEET SUGAR

CHAPTER II

Purchase and Analysis of Beets	48
Saccharimetric Tables	63

CHAPTER III

Preliminary Treatment of the Beets	67
--	----

CHAPTER IV

Diffusion—Beet Slicers—Knives—Diffusion Batteries—The Utilisation of Exhausted Pulp—Pulp Eliminators—The Chemical Control of Diffusion	77
--	----

CHAPTER V

	PAGE
Defecation and Carbonatation	117
Horsin-Deon's and Guibert's Systems	123
Chemical Analysis and Filtration of the Limed Juice	125
The Chemical Control of the Carbonatation Process	127
Manufacture of Carbonic Acid and Quicklime	142

CHAPTER VI

The Exhaustion and Utilisation of the Sludge Filtration	152
Filter Presses and other Filters	153
Washing and Revivifying of Animal Charcoal	167
Disposal of Spent Char	170

CHAPTER VII

The Concentration of the Beet Juice to Syrup	177
Single and Multiple Effect	180
Evaporation <i>in Vacuo</i>	180
Area of Heating Surfaces	181
Thermo-dynamical Data and Formulæ and their Practical Application	182
Motor Power of French Beet-sugar Factories	185
The Steam consumed per Hectolitre of Juice	187
Method of working Triple-effect Plant	193
Froth Preventers and Sugar Recoverers	216
Condenser and Air-Pumps	220
The Pressure in the Condenser	223
Quadruple and Quintuple Evaporation	224
Utilisation of the Multiple-effect Steam in Reheating the Juice, and the Saving thus effected	225
Fuel Consumption in Austrian Factories	230
The Ammoniacal Vapours and Pumps	231
Elimination of Ammoniacal Gases	231
Cleaning the Apparatus	232
Removing Encrustation	232
Horizontal Systems of Multiple-effect Evaporation	233
The Yaryan Streaming Process	233
Automatic Sampling	234

CHAPTER VIII

Evaporation in Vacuum Pans	236
The Treatment of Weak and Bad Quality Liquors	236
The Point of Saturation of Sugar Solutions at various Temperatures	236
Tables	236

CONTENTS

ix

	PAGE
Boiling in Grain	240
The Radiating Heater	242
The Bocquin-Lepchinsky Cooler	243
Crystallisation in Motion	243
Storing and Cooling Tanks	243
Tip-tilting Waggon Tanks	244
Analyses of Syrups of <i>Masse Cuite</i>	244
Difficulties in Boiling	247
Turbines	249
Cleansing the Sugar	256
Brown Sugar	260
Analyses of Syrup and <i>Masses Cuites</i>	261
Influence of Purity of the <i>Masse Cuite</i> on the Yield of Sugar	264
Sugar Warehouses	264
Beet-sugar Molasses	264
Extraction of Sugar from Molasses	265
The Old Alcoholic Method	265
Dubrunfaut's Osmogènes	266
Evaporating Osmogènes	267
Sucrate of Lime Process	268
Manoury's Alcoholic Method	270
The Strontia Method	271
Advantages of Sucrate of Strontia over Sucrate of Lime	271
Continuous Sulphitation of the Syrup and Drainage Liquors	272
Attempts to abolish Molasses	273
Crystallisation in Motion	274
Steffen's Process	275
The Simplex Process	277
Ragot and Tournour's Process	279
Cost of Manufacture	280

SECTION III

CANE SUGAR

CHAPTER IX

The Sugar Cane	282
The Sugar Cane and its Average Richness in Sugar	282
The Composition of the Sugar Cane	282
Colouring Principles	284
Cane Sugar Wax or Cerosin	284
Culture of the Sugar Cane	284
The Yield of Sugar per Ton of Cane	288
Extraction of the Juice by Milling	290
Modern Mills	291

	PAGE
Cane Conveyors	292
The Mechanics of the Cane Mill	292
Speed of Rolls	294
Machine for forcing the Juice from the Cane by Percussion	295
Exhaustion of the Bagasse by Systematic Washing	296
Shredders	296
Systematic Washing	297
Perichon's Process	298
Bagasse as Fuel	299
Furnace for burning Green Bagasse	300

CHAPTER X

Sugar-Cane Diffusion—The Results obtained on the Large Scale by the United States Department of Agriculture at Magnolia, Louisiana, U.S.A. (an Elaborate Report)	301
--	-----

CHAPTER XI

Defecation of the Cane Juice	323
Cane Juice and its Properties	323
Eliminating Impurities	324
Sulphitation	326
Evaporation to Crystallisation-point in Vacuum Pans	328
Compact Plant for Colonial Use	330

SECTION IV

SUGAR REFINING

CHAPTER XII

The Raw Material	331
White Sugar	331
Yellow Sugar	331
Brown Sugar	331
Purchase and Selection	331
Typical Analyses of different Brands of Raw Sugar	332
A Product of Predetermined Quality to be aimed at and Work regulated accordingly	333
Preliminary Turbinizing	334
Solution by Water and Steam	335
Defecation	336
Filtration	339
Taylor's Filters	339

CONTENTS

xi

	PAGE
Animal-Charcoal Filters	341
Impurities of the Carbon	342
Function of the Nitrogen	343
Injurious Action of Free Ammonia and Ammonium Sulphide in Char	344
Presence of Interstitial Occluded Gases in Animal Charcoal	344
Substitutes—Basic Acetate of Lead and Sulphurous Acid	344
Decolorisation by Ozone	345
Defecation of Cane Sugar by the Double-Carbonatation Process	345
Size or Grist of the Granulated Char	347
Loss of Sugar in the Bone-black Room	348
Revivifying Animal Charcoal	349
Change in Composition on repeated Re-burning and Re-use of Animal Charcoal	351
Method of Removal of Mineral Salts	351
Bad Effect of permanently Hard Water	352
Old and New Charcoal, Ratio of Bulk to Gravity	353
Effects of the Quality of the Water on the Charcoal	354
Treatment of the Charcoal with Dry Hydrochloric Acid	355
Precautions to be observed on Estimating the relative Decolorising Power of Chars	355
Boiling the Sugar in Vacuum Pan	356
Boiling-point	356
Howard's Vacuum Pan	357
The Steam Jacket	357
Coil Barometers, Thermometers, and Feeding Apertures	358
The Wet Air-Pump	359
Injection Condenser	360
Instructions for using the Vacuum Pan	361
Precautions in applying Steam	362
Heating Power of High and Low Pressure Steam	364
Detecting Leakage	365
Measuring the Liquor	366
The Capacity of the Condensers	366
Decrepitation in Pan as Crystals begin to form	367
<i>The Flash</i>	368
<i>Taking the Proof</i>	368
Examination of the Proof	369
Feeding the Pan	369
Final Precautions	370
Boiling by Doubles	371
Boiling by the Thread Proof	371
Cleansing of the Proof-stick	372
Reheaters	373
The Fill-house	376
The Making of Sugar Loaves	376
Liquoring	378
"Magna Liquor"	378

	PAGE
Fine Liquor	378
The Facing Machine	379
Facilitating Percolation and Drainage by Suction	380
Turbining Sugar Tablets	380
Raw Syrup or Molasses	381

SECTION V

SELECTION OF SUGARS

CHAPTER XIII

The Chemistry of Molasses, Invert Sugar, and Raffinose	382
The Analyses of Molasses and Table Syrups	383
Clerget's Table	385
Estimation of Raffinose	386
Composition of Table Syrups	388

CHAPTER XIV

The Analyses of Raw Sugars	390
Commercial Glucose and Glucose Syrups	394

CHAPTER XV

The Estimation of Sugars in Wine, Diabetic Urine, Milk, Honey, and Jam	396
--	-----

INDEX	403
-----------------	-----

LIST OF ILLUSTRATIONS

FIG.	PAGE
1. Haensch's Saccharimeter	17
2. Polarisation Discs	18
3. Zeiss's Portable Saccharimeter	21
4. Pellet's Beet Pulper	57
5. Drill for Sampling Beets	57
6. Hanriot's Beet Pulper	58
7. Apparatus for Continuous Saccharimetrical Analysis	61
8. Fricourt's Machine for Washing Beets	69
9. Stone Eliminator	70
10. Beet-wiping Machine	72
11. Beet Shaker	72
12. Beet Elevator	74
13. Circular Diffusion Battery	82
14A-B. Slicing Knife and Knife Holder	88
14C-D. Knife Holder and Stone Remover	89
15. Fontane and Rassman's Slicer	90
16. Rotary Drum Slicer	91
17. " " on old body	92
18. " " ordinary body	93
19. " " " "	94
20. Slice-sampling Apparatus	95
21. Circular Diffusion Battery	97
22. Feeding Diffusion Batteries	98
23. Feeding and Clearing Circular Battery	99
24. Cail's Diffuser	100
25. Air-removing Apparatus	102
26. Bergneen's Exhausted Pulp Press	105
27. Pfeiffer's Automatic Diffusion Process	113
28. Pellhardt's Pulp Eliminator	115
29. Juice-liming Vessels	126
30. Intermittent Carbonatation Tanks	128
31. Horsin-Deon's Automatic Register	133
32. Automatic Juice-sampling Apparatus	134
33. Lungen's Automatic Register	135
34. Horsin-Deon's Continuous Carbonator	138
35. Gibert's Continuous Carbonatation Process	140
36. Radiating Heater in use	141
37. Lime Kiln and Gas Scrubber	145
38. Sidersky's Twin Calcimetric Burettes	148

THE TECHNOLOGY OF SUGAR

CHAPTER I

THE CHEMISTRY OF SUCROSE, LACTOSE, MALTOSE, GLUCOSE, LEVULOSE, AND INVERT SUGAR; AND THEIR SACCHARIMETRICAL, VOLUMETRICAL, AND GRAVIMETRICAL DETERMINATION

DEFINITION.—Sugars are substances with a sweet taste which are soluble in water, and capable of being decomposed into carbonic acid and alcohol by a peculiar chemical reaction induced by the action of a ferment (yeast). This change of sugar into alcohol, etc. is known as the alcoholic or vinous fermentation.

All sugars belong to an important class of organic bodies called the *carbohydrates*, a name which does not mean that they contain water, but that they contain carbon, hydrogen, and oxygen, and that the hydrogen and oxygen are present in the same proportion as in water.

They may be divided into two classes—

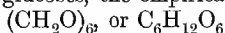
1. The *glucoses* or *monoses*,—those analogous to grape sugar (glucose), $C_6H_{12}O_6$.
2. The *saccharoses* or *bioses*,—those analogous to cane sugar (sucrose), $C_{12}H_{22}O_{11}$.

1. *Grape* sugar (glucose), the principal member of the first group, is the principal constituent of the juice of the grape and other sweet fruits. Honey also contains much grape sugar.

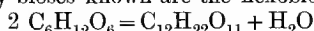
2. *Cane* sugar (sucrose) and *milk* sugar (lactose) are the chief sugars of the second group. Cane sugar is got from the juice of—(1) The sugar cane; (2) the sugar beet; (3) the sugar maple; (4) the sugar palms, and (5) is also present in most sweet fruits associated

with sugars belonging to group 1. Milk sugar is present in the milk of all mammals, and is prepared by evaporating down the whey and letting the sugar crystallise out.

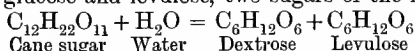
The *monoses* are compounds, the functions and behaviour of which are complex; they behave at the one time as polyatomic alcohols, at other times as aldehydes or ketones. By hydrogenation they reproduce a polyatomic alcohol. The most important members are the hexamonomoses or glucoses, the empirical formula of which is—



The *bioses* are produced by the combination of two monoses, with loss of water. They may be likened to the oxide ethers of the monoses. The only bioses known are the hexobioses or *saccharoses*—



If we boil *cane* sugar with a dilute acid it takes up water and is converted into glucose and levulose, two sugars of the first group—

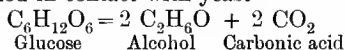


Milk sugar is acted on in the same way.

Differentiation of the monoses from the bioses; difference in stability under action of reagents, ferments, etc.—These two classes of sugars are principally differentiated from each other by their different susceptibility to alteration in their chemical constitution; the second group, *i.e.* the bioses or cane-sugar group, being more stable than the first or monose group, *i.e.* the monoses.

Bioses do not directly ferment.—The bioses, for instance, do not ferment directly; they are only capable of undergoing the alcoholic fermentation after having first fixed the elements of water, and thus become changed into sugars of the grape-sugar class, and then the latter ferment.

Direct fermentescibility and general instability of the monoses.—On the other hand, the sugars of the grape-sugar class easily undergo alcoholic fermentation in contact with yeast—



Moreover, they begin to be decomposed when treated with caustic alkalis in the cold, and at 100° C. (212° F.) the reaction is complete. They reduce cupro-potassic solutions in the cold, or at least at 100° C.

The reagents which decompose monoses have little or no action on the bioses.—But caustic alkalis have little or no action on sugars belonging to the cane-sugar group at 100° C. (212° F.), and at that temperature they have only a feeble action upon cupro-potassic solutions.

Sugar of the cane-sugar classes (bioses) crystallise readily, those of the monose class with difficulty.—The two classes of sugars are further distinguished from one another by the readiness with which cane sugar crystallises, whilst not only is grape sugar difficultly and

imperfectly crystallisable, and is usually obtained in a granular state, but it is *much inferior in sweetness*.

CANE SUGAR

Properties.—Sugar crystallises in oblique, rhomboidal prisms (sugar candy) with hemihedral facets. The crystals are hard and anhydrous, and phosphoresce when rubbed or crushed in the dark. Their density is equal to 1·606. They are permanent in the air.

Taste.—Sugar possesses a well-known and characteristic sweet taste. Hence the adage “sweet as sugar,” in contradistinction to “bitter as gall.”

I.—DENSITY OF SOLUTIONS OF SUGAR OF DIFFERENT STRENGTHS AT 17°·5 C.*

Per Cent.	Density.		Per Cent.	Density.		Per Cent.	Density.	
	Balling.	Niemann.		Balling.	Niemann.		Balling.	Niemann.
1	1·0040	1·0035	26	1·1106	1·1103	51	1·2385	1·2378
2	1·0080	1·0070	27	1·1153	1·1150	52	1·2441	1·2434
3	1·0120	1·0106	28	1·1200	1·1197	53	1·2479	1·2490
4	1·0160	1·0143	29	1·1247	1·1245	54	1·2553	1·2546
5	1·0200	1·0179	30	1·1295	1·1293	55	1·2610	1·2602
6	1·0240	1·0215	31	1·1343	1·1340	56	1·2667	1·2658
7	1·0281	1·0254	32	1·1391	1·1388	57	1·2725	1·2714
8	1·0322	1·0291	33	1·1440	1·1436	58	1·2783	1·2770
9	1·0363	1·0328	34	1·1490	1·1484	59	1·2841	1·2826
10	1·0404	1·0367	35	1·1540	1·1533	60	1·2900	1·2882
11	1·0446	1·0410	36	1·1590	1·1582	61	1·2959	1·2938
12	1·0488	1·0456	37	1·1641	1·1631	62	1·3019	1·2994
13	1·0530	1·0504	38	1·1692	1·1681	63	1·3079	1·3050
14	1·0572	1·0552	39	1·1743	1·1731	64	1·3139	1·3105
15	1·0614	1·0600	40	1·1794	1·1781	65	1·3190	1·3160
16	1·0657	1·0647	41	1·1846	1·1832	66	1·3260	1·3215
17	1·0700	1·0693	42	1·1898	1·1883	67	1·3321	1·3270
18	1·0744	1·0738	43	1·1951	1·1935	68	1·3383	1·3324
19	1·0788	1·0784	44	1·2004	1·1989	69	1·3445	1·3377
20	1·0832	1·0830	45	1·2057	1·2043	70	1·3507	1·3430
21	1·0877	1·0875	46	1·2111	1·2098	71	1·3570	1·3483
22	1·0922	1·0920	47	1·2165	1·2153	72	1·3633	1·3535
23	1·0967	1·0965	48	1·2219	1·2209	73	1·3696	1·3587
24	1·1013	1·1010	49	1·2274	1·2265	74	1·3760	1·3658
25	1·1059	1·1056	50	1·2329	1·2322			

* Cp. Scheibler's table, p. 64.

Solubility.—Sugar dissolves in one-third of its weight of cold water. The resulting solution is thick and viscous, and is known as

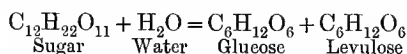
simple syrup. A solution of 100 parts of sugar in 50 parts of water (*i.e.* 20 lbs. sugar to 1 gallon of water) shows a density of 1·345 at 15° C., and marks 37° by Baumé (69° Tw.) The lbs. of sugar in a gallon of syrup may be approximately got by multiplying the decimal part of the gravity by 26 (Ure).

Solubility in alcohol.—Sugar is insoluble both in ether and in cold absolute alcohol. Boiling absolute alcohol dissolves about 1¼ per cent. Boiling ordinary alcohol dissolves more. Sugar is deposited from its alcoholic solution on cooling in the form of small but distinct crystals.

II.—SOLUBILITY OF CANE SUGAR IN MIXTURES OF ALCOHOL AND WATER.

Percentage of Alcohol.	At 0° C.		At 14° C.		At 40° C.
	Density at 17°·5 C.	Sugar Per Cent.	Density at 17°·5 C.	Sugar Per Cent.	Sugar Per Cent.
		Grammes.		Grammes.	
0	1·3248	85·8	1·3258	87·5	105·2
10	1·2991	80·7	1·3000	81·5	95·4
20	1·2360	74·2	1·2662	74·5	90·0
30	1·2293	65·5	1·2327	67·9	82·2
40	1·1823	56·7	1·1848	58·0	74·9
50	1·1294	45·9	1·1305	47·1	63·4
60	1·0500	32·9	1·0582	33·9	49·9
70	0·9721	18·2	0·9746	18·8	31·4
80	0·8931	6·4	0·8953	6·6	13·3
90	0·8369	0·7	0·8376	0·9	2·3
97·4	0·8062	0·08	0·8082	0·36	0·5

Action of ferments.—Sugar does not ferment directly, but in the presence of the germs present in yeast and also of the peculiar ferments contained in most saccharine fruits it fixes water and is converted into a mixture of dextrose and levulose known as invert sugar—



This transformation is also effected when sugar is boiled for a long time in water. Hence the precaution which is taken in the manufacture of sugar to evaporate the clarified juices at a low temperature.

Action of acids—Invert sugar.—Under the action of dilute acids sugar is likewise changed into invert sugar. The change takes place slowly in the cold, but rapidly on boiling. Dilute sulphuric acid is more effectual in its action, in this respect, than other, and more especially organic, acids. The original solution deviates the plane of polarisation to the right. After treatment with dilute acids, and

cooling, the liquid deviates to the left, for the rotary power of levulose to the left is greater than that of glucose to the right. Organic acids produce the same effect on boiling, but at the ordinary temperature their action is extremely slow—which explains the co-existence of cane sugar and of acetic, malic, and tartaric acids in certain fruit sugars. When sugar is subjected to prolonged ebullition with dilute sulphuric or hydrochloric acids the solution eventually becomes brown, and ulmic substances are formed.

Organic acids of the fatty series (C^nH^{2n+1})COHO, such as acetic, butyric, or stearic acids, combine with sugar at 120° C., forming neutral bodies analogous to oils and fats; the rotary power does not decrease on keeping the solution, nor even when it is boiled.

Boiling glacial acetic acid transforms cane sugar in a few minutes into an acetic ether.

Concentrated sulphuric acid carbonises sugar rapidly. The action is rapid, generates heat, and produces an evolution of sulphurous acid gas.

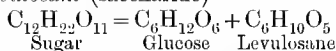
Concentrated nitric acid converts sugar into oxalic acid. A mixture of bichromate of potash, peroxide of manganese, and sulphuric acid energetically oxidise sugar with production of formic acid.

By adding powdered sugar to a mixture of nitric and sulphuric acids, cooled to 2° C., a nitrated product is obtained, which would appear to consist of tetranitric saccharose. It forms an amorphous mass, which detonates on concussion.

Dry chlorine does not act on dry sugar. Chlorine attacks sugar syrup at a temperature of 100° C., and forms hydrochloric acid and brown compounds. Certain chlorides, such as antimonic and stannic chlorides, impart a brown coloration to sugar.

Action of heat.—Sugar melts at 160° C. into a thick transparent liquid, which on cooling is transformed into an amorphous vitreous mass (*barley sugar*); but gradually, in consequence of molecular change, the mass again becomes crystalline.

When sugar is kept for a long time at 160° to 161° C. it splits up into *glucose* and *levulosane* (saccharide)—



When this mixture of glucose and levulosane is dissolved in water and fermented the glucose first disappears, and the levulosane remains behind and may be isolated by evaporating the solution and heating the residue to 170° C.

Caramel.—When sugar is heated from 190° to 220° C. it continuously loses water, and is converted into a brown, amorphous, bitter substance, soluble in water, known under the name of caramel.

Dr. Prout found that sugar exposed for seven hours to a temperature of 300° F. only lost 0.6 per cent. of its weight, but its properties

the glucose in cold water, it may be dissolved in hot 90 per cent. alcohol. It crystallises more easily from alcohol. When the diabetic urine is not very rich in glucose, it deposits, in addition to crystals of the latter, crystals consisting of a combination of glucose or common salt, or even these alone.

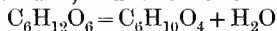
Properties.—Glucose is generally met with in the form of mammillary white opaque crystals—agglomerated after the manner of a cauliflower, containing two equivalents of water of crystallisation. They are permanent in the air. They soften at 60° C., melt in the water bath, and lose their water of crystallisation at 100° C., or even in a current of air at 80° C. Glucose is deposited from its alcoholic solution in anhydrous needles, which do not melt until 196° C. Glucose is three times less soluble in water than cane sugar, and its solution of equal strength is three times less sweet. One part of glucose dissolves in 1.2 of water at 17° C. Glucose dissolves very easily in dilute alcohol, but is less soluble in absolute alcohol. Anthon has determined (*Chemisches Centralblatt*, Bd. v. S. 292) that glucose is soluble in alcohols of different strengths, at 64° F., to the following extent—

	Specific Gravity.				
100 parts of alcohol	0.837	will dissolve	1.95	parts of glucose.	
”	0.880	”	9.30	”	”
”	0.910	”	17.74	”	”
”	0.950	”	36.45	”	”

After some time, however, a little glucose will crystallise from these solutions; and the following numbers, the author says, will show the real solubility—

	Specific Gravity.			Per Cent.	
Alcohol	0.837	will hold in solution	1.94	of glucose.	
”	0.880	”	8.10	”	”
”	0.910	”	16.00	”	”
”	0.950	”	32.50	”	”

Action of heat on glucose.—When glucose is heated to about 170° C. it melts, loses water, and is converted into glucosane—



Glucosane has not yet been obtained in a pure state. It does not ferment directly, but ebullition with acids changes it into fermentable glucose. When glucose or glucosane are heated to high temperatures they are changed into brown products analogous to caramel.

Action of acids.—Glucose is not coloured when treated in the cold by concentrated sulphuric acid; it combines therewith, forming a sulphoglucosic acid analogous to sulphosaccharic acid. But if the temperature rises the glucose is charred with evolution of sulphurous acid. Dilute boiling nitric acid converts glucose into oxalic acid.

Action of bases.—Alkaline bases decompose glucose slowly at first, rapidly on boiling. If a solution of caustic potash, e.g., be added to

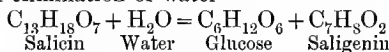
a solution of glucose, the liquid turns yellow as soon as heat is applied, afterwards turning a very deep brown. The same coloration is produced when glucose is heated with caustic baryta or caustic lime. This reaction is so delicate as to enable it to be used in the detection of glucose when used as an adulterant of cane sugar in commercial products; glucose combines with the alkaline earths and lead oxide to form crystalline products. It combines with caustic baryta to form a *glucosate of baryta* ($\frac{1}{2}$ $C_6H_{12}O_6$, 3 BaO + 4 H_2O), which is obtained as a white crystalline powder when an alcoholic solution of glucose is mixed with a solution of barium hydrate in weak alcohol. A basic glucosate of lead ($C_6H_{12}O_6$, 3 PbO + 2 H_2O) is precipitated when ammonia is added to a mixed solution of glucose and lead acetate. When slaked lime is dissolved in a solution of glucose and the solution mixed with alcohol a white precipitate of glucosate of lime is formed.

The reducing action of glucose is discussed elsewhere.

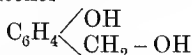
Glucosic ethers.—Glucose behaves with acids like an alcohol; it, in fact, forms with acids, with elimination of 1, 2, 3, 4, or even 5 molecules of H_2O , true ethers, the glucosides of Berthelot. These glucosides are obtained by heating glucose in sealed tubes with an acid, but mineral acids exert a destructive action on glucose at temperatures between 100° and 120° C., the temperatures at which these reactions take place. Glucosides, therefore, are not easily prepared, except those from organic acids (tartaric, acetic, and stearic acids). By dissolving glucose in fuming nitric acid the pentanitric glucoside is prepared, which is sufficient to establish its pentatomic alcoholic nature. All these glucosides split up in presence of dilute acids into glucose and the producing acid. Some of the glucosides of organic acids exist in acid fruits.

Glucosides.—A great number of neutral crystalline principles are met with in plants which split up like the above-mentioned glucosides into a fermentable glucose; but one of the products is an alcohol or a phenylic alcohol.

To this class belong salicin, the bitter principle contained in different species of elm, poplar, esculin contained in the bark of the purging nut tree, coniferin extracted from conifers. These neutral glucosides may be regarded as the result of the union of glucose with an alcohol, with elimination of water—

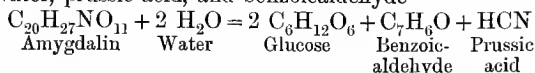


Saligenin is a phenylic alcohol—



The saccharoses themselves, although their synthesis cannot be effected from two glucoses, also belong to the same class, by the manner in which they split up.

Moreover, glucosides are known which split up by absorbing the elements of water into a glucose, an acid, an alcohol, or an aldehyde. Such is *amygdalin*, contained in bitter almonds and the almonds of a number of stone fruits, and which yields, by decomposition in presence of water, prussic acid, and benzoic aldehyde—



The above is the reaction by which the essential oil of bitter almonds is produced from amygdalin.

LEVULOSE (FRUCTOSE, FRUIT SUGAR) ($\text{C}_6\text{H}_{12}\text{O}_6$)

Levulose—this substance often termed uncrystallisable fruit sugar—forms the uncrystallisable portion of the saccharine matter of certain fruits (raisins, cherries, gooseberries), in which it is present along with glucose, and generally to the same extent, just as if they resulted from the inversion of cane sugar by the action of acids. It also forms the uncrystallisable portion of honey.

By heating cane sugar with dilute acids it is changed into a mixture of equal weights of dextrose and levulose; this mixture is known as *invert sugar*. Levulose in a pure state is obtained very rapidly by saccharifying inulin with dilute acids. (Inulin is a substance isomeric with starch, contained in the roots of elecampane, dahlia, colchicum, and artichoke.) Levulose may be isolated from *invert sugar* by mixing 10 grammes of the latter with 6 grammes of slaked lime. The mass, which is at first liquid, becomes pasty on stirring. It then contains liquid glucosate of lime and solid levulosate of lime. It is put into a cloth and then subjected to pressure. The solid part remaining behind is the calcareous salt of levulose, all the glucose having been pressed out as glucosate of lime. The solid calcareous salt is dissolved in water and decomposed by a current of carbonic acid. The filtered and evaporated solution yields pure levulose.

Pure levulose.—Into a 50 c.c. *Erlenmeyer* flask put 50 c.c. water and 5 c.c. HCl, of density to give 0.01 to 0.02 per cent. HCl to weight of inulin. (For 200 grammes inulin of 0.25 per cent. ash content, 5 c.c. normal HCl; for higher ash content, more.) The 200 grammes of inulin, in fine powder, are then added and well shaken and heated in boiling water (not in bath). After thirty minutes from time mass is evenly heated the maximum of the inversion is reached, the flask is removed from the boiling water. The syrup, carrying some granules of inverted inulin, is poured into 1 litre of absolute alcohol, a little blood char added, and allowed to stand for twenty-four hours, and filtered. A few crystals of fruit sugar added to the filtrate cause a copious crystallisation of pure levulose.

A better yield is secured by evaporating the alcoholic filtrate to a thick syrup and placing over H_2SO_4 in dessicator. A crop of crystals is thus obtained, which resemble dextrose got in similar conditions. The crystal masses thus obtained are dissolved in 3 to 4 parts absolute alcohol, allowed to stand twelve hours, and the clear solution poured off. A few crystals of fruit sugar and rubbing with a glass rod cause the crystallisation of the levulose to appear at once.

Solubility.—Levulose when pure is an uncrystallisable syrup, with a much sweeter taste than glucose syrup. It dissolves very readily in water and in ordinary alcohol, but not so easily in absolute alcohol.

Rotary power.—It is levo-rotary, and its power is -106° at $15^\circ C.$, but it varies greatly with the temperature: thus at $90^\circ C.$ it is decreased by one-half, becoming -53° .

Glucose, on the other hand, having a dextro-rotary power which does not vary with the temperature, the variations in the rotary power of levulose ought to be still present in invert sugar, which is a mixture of equal parts of glucose and levulose. As a matter of fact, inverted sugar whose rotary power is -25° at $15^\circ C.$ is only half as much at $52^\circ C.$, is nil at $90^\circ C.$, and changes its sign above that temperature.

Action of heat.—Levulose begins to decompose above $100^\circ C.$, and gives the same products of decomposition as glucose. With lime it forms an insoluble compound, $2 C_6H_{12}O_6, 3 CaO$.

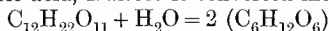
Although levulose decomposes much more readily under the influence of acids or of heat, it, however, resists the action of alkalies or ferments much better. In fact, its greater resistance to the action of ferments has been utilised in its preparation. If during the course of fermentation the rotary power of the fermenting liquid be taken from time to time, it will be found, at the end of a certain time, that the deviation to the left attains its maximum and then diminishes; on arresting the fermentation at this point the glucose is destroyed, and the liquid only contains levulose.

MALTOSE

Maltose is produced when amylaceous bodies are heated at $60^\circ C.$ with water and germinated barley (malt). Under the influence of the *diastase* the amylaceous matter is transformed into dextrine and then into maltose—



Crystallised maltose has the formula $C_{12}H_{22}O_{11} + H_2O$. Heated with dilute sulphuric acid, maltose is converted into glucose—

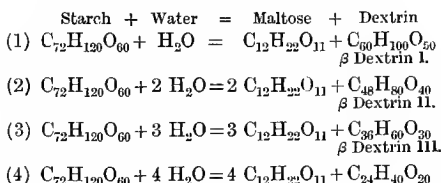


Maltose is dextro-rotary like glucose, but to a far greater extent

than ordinary glucose (see Tables III. and VI.). It reduces cupropotassic solutions, although not so easily as the glucoses. It ferments under the same conditions as cane sugar.

Maltose sugar was first discovered by *Dubrunfaut*, but it was forgotten until *O'Sullivan* of Burton-on-Trent again studied the action of albumenoid ferments, such as are to be found in malt upon starch. The following table represents the reaction—

HYDRATION PRODUCTS OF STARCH



The first action is to add 1 molecule of water, which is effected by the affinity of albumenoid ferments. The same action is also brought about by the saliva of man, because that also contains an albumenoid ferment called *ptyaline*. In malt there is a similar ferment, *diastase*. When the first molecule of water is added it causes a lesion of the complex starch molecule into the distinct product maltose sugar ($\text{C}_{12}\text{H}_{22}\text{O}_{11}$) (the isomer of cane sugar and 5 molecules of dextrin— $\text{C}_{12}\text{H}_{20}\text{O}_{10}$). The next stage is to add 2 molecules of water; then a further lesion is brought about by the ferment. In the last stage 4 molecules of water are added, and thus 4 molecules of maltose sugar and 2 of dextrin are produced. *Brown* and *Heron* and *Gruber* and *Musculin* give some other hydration products in addition to those given by *O'Sullivan*.

Although it presents many analogies with cane sugar, yet maltose may be differentiated from the former by the fact that it is precipitated from its aqueous solution by alcohol, whilst cane sugar is not precipitated under similar conditions. Alcohol only dissolves maltose in minute proportions. Maltose also differs from cane sugar in the following respects. The formula is the same, that is, the composition of 100 parts is the same, and the molecular weight is perhaps the same, but the properties that distinguish maltose are the following. Cane sugar rotates the plane of polarised light to the right, maltose rotates the ray also to the right, but about twice as far. It is much more dextro-rotatory than cane sugar and glucose, although less so than starch. *Fehling's* solution has no action on cane sugar, except, however, after a time, when the alkali begins to break up the sugar. It has no immediate action; but on maltose sugar *Fehling's* solution produces an abundant and immediate reaction on boiling; still, the amount of reduction of the protoxide of copper to suboxide is not so great as in the case of dextrose and levulose sugars.

LACTOSE OR MILK SUGAR ($C_{12}H_{22}O_{11} + H_2O$)

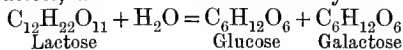
History.—This substance was first described by *Bartholetti* (*Encyclopædia Hermatico-dogmatica*, Bologna, 1619, "Manna, seu nitrum seri lactis"). But the person who contributed to make sugar of milk generally known was *Ludovico Festi*, who gave it out as an invention of his own, and sold it as a remedy in gout and other diseases. He was a physician in Venice, where he died in 1707. After his death *Valisneri* published the process which Festi employed in extracting this sugar from whey.

Preparation.—Lactose or milk sugar can only be obtained by evaporating the whey obtained as a liquid by-product in cheese-making. It is said to have at one time been largely manufactured as an article of food in Switzerland. It may be prepared by coagulating the milk by a small quantity of sulphuric acid. The whey, filtered, evaporated, and the syrupy solution allowed to stand in an open place, deposits small but very hard crystals, which are purified by fresh crystallisation and bleaching by animal charcoal. It is met with in commerce in cylindrical pieces consisting of an agglomeration of crystals around a wooden stick serving as an axis.

Properties—Crystalline form.—Milk sugar crystallises in right rhomboidal prisms terminating in octahedral points. *Density*—1.53. It is hard, transparent, and gritty between the teeth. Its taste is but very slightly sweet. On keeping, it becomes covered with moulds. It dissolves in 6 parts of cold water with evolution of heat and in $2\frac{1}{2}$ parts of boiling water. Cold alcohol and ether do not dissolve lactose. Alcohol therefore precipitates lactose from its aqueous solution. Crystals of lactose dried at 100° C. have the formula $C_{12}H_{22}O + H_2O$. At 150° C. they lose their water of crystallisation, and have then the same centesimal composition as cane sugar. At this temperature the crystals, in fact, begin to decompose. The dehydrated crystals become brown without melting at 160° C.; at 175° C. they change into *lactocaramel* ($C_{12}H_{20}O_{10}$), an amorphous brown tasteless substance. The sugar of milk does not melt until 203.5° C.

A solution of sugar of milk is dextro-rotary. Its rotary power referred to the formula $C_{12}H_{22}O_{11}$ is equal to 59.3 . It is greater by .38 in recent solutions, but soon diminishes to the constant term.

Like glucose, it reduces cupro-potassic solutions, even in the cold. Its reducing power is less than glucose, its cupreous precipitate being only $\frac{1}{16}$ that of the latter, other things being equal. When lactose is boiled with dilute mineral acid it splits up into two glucoses, ordinary glucose and *galactose*, which is also dextro-rotary—



Action of acids.—Lactose is carbonised at 100° C. under the

graduated to hold 100 grammes of water at $17^{\circ}5$ C. has the following relation to a true 100 c.c. flask, namely 100.12 : 100 c.c. Instead of a true 100 c.c. flask at this temperature holding 100 grammes as a Mohr flask does, it holds exactly 99.8747 grammes of water. A certificate should be obtained from the maker stating in the case of each instrument the method of its graduation, and then use with that instrument the same flask and same weights which were employed in obtaining this graduation. If the instrument be used with a Mohr flask containing 100 grammes of water weighed in air at a temperature of 4° , then all flasks should be graduated in this way, whether they be weighed in air or *in vacuo*. In the same way, if the standard weights of sugar used in graduating the instrument are weighed in air, we should make our weighings in air; if, however, the standard weights of sugar are corrected for weight *in vacuo*, then we should also correct for weight *in vacuo*.

III.—GRADUATION AND ADJUSTMENT OF POLARISCOPIES: FRENCH AND GERMAN SCALES (SIDERSKY).

Substance.	Specific Rotary Power.	French (A).		French (B).		German.	
		Official Formula.		New corrected Formula.		Ventzke's Scale.	
		Metric Graduation.	Mohr's Graduation.	Metric Graduation.	Mohr's Graduation.	Metric Graduation.	Mohr's Graduation.
	grms.	grms.	grms.	grms.	grms.	grms.	grms.
Cane sugar	66.51	16.19	16.23	16.29	16.33	26.000	26.048
Glucose (dextrose)	52.74	20.40	20.45	20.49	20.54	32.765	32.820
Lactose (milk sugar)	52.53	20.51	20.56	20.60	20.65	32.970	33.025
Maltose	138.30	7.78	7.80	7.82	7.84	12.510	12.520
Soluble starch and dextrin	194.80	5.50	5.51	5.51	5.53	8.835	8.850

1 French degree (Laurent's type) = 0.6277 Ventzke = 0.2167 arc degrees.

1 German degree (Ventzke's type) = 1.5931 Laurent = 0.3440 arc degrees.

To describe all the various forms of polariscopes would only confuse the reader; we shall therefore confine ourselves to one single type of instrument, namely, the half-shadow instrument made by Franz Schmidt & Haensch of Berlin (Figs. 1 and 2), which may be used with white light from either paraffin oil or gas lamps. It is handy to manipulate, and its indications are easily read, even by one who is colour-blind. This form of instrument is adjusted to the Ventzke scale, in which 1° = the $\frac{1}{100}$ part of the rotation produced in the plane of polarisation of white light in a column 200 millimetres long by a standard solution of chemically pure sucrose at $17^{\circ}5$ C., the standard solution of sucrose in distilled water being such as to contain at $17^{\circ}5$ C., 26.048

grammes of sucrose in 100 c.c., the weights and volumes being considered as absolute, all weighings being referred to a vacuum. The above definition should be supplemented with a statement of the equivalent circular rotation in degrees, minutes, and seconds that would be produced by the standard solution of sugar used to read 100° on the scale. The instruments should be adjusted by means of control quartz plates, three different plates being used for complete adjustment—one reading approximately 100° on the scale, one 90° , and one 80° . These control plates should have their exact value ascertained, in the terms of the Ventzke scale, by comparison with the standard quartz plates, in strict accordance with the foregoing definition, and should also be accompanied by tables giving their values from 10° to 35° C.

The *weights* used should be of solid brass, and should be standardised. The *flasks* should be of such a capacity as to contain at 17.5° C. 100.06 c.c. when filled, in such a manner that the lowest point of the meniscus of the surface of the liquid just touches the graduation mark. The flasks are standardised to contain this volume, in order that the results shall conform to the scale recommended for adoption without numerical reduction of the weighings to *vacuo*. They should be standardised. *Tubes*.—These should be of brass or glass, 200 and 100 millimetres in length, and should also be standardised. *Balances* should be sensitive to 1 milligramme.

Arrangement of laboratories for polarisation work.—This has an important bearing on the accuracy of the results. The reading is more readily and accurately taken if the eye of the observer be screened from diffused daylight (as in Fig. 7), therefore a partial darkening of the room, which may be accomplished by means of curtains or hangings, is an advantage. On the other hand, the temperature at which the observation is made also affects the results, so that the arrangements for darkening the room must not interfere with its proper ventilation; otherwise the heat from the lamps used, if confined within a small room, will cause considerable variations in the temperature of the room from time to time.

The proper conditions will possibly best be met by placing the lamps either in a separate room from that in which the instruments are and perforating the wall or partition between the two rooms for the light to reach the end of the instruments, or in a ventilated hood with the walls perforated in a like manner. By lining the wall or partition on both sides with asbestos paper, and inserting a plate of plane glass in the aperture through which the light passes, the increase of temperature from the radiation of the lamp will be still further avoided. With the lamps separated from the instruments in this manner, the space in which the instruments are contained is readily darkened without much danger of its temperature being unduly raised.

Some light, of course, is necessary for reading the scales, and if

artificial light is employed for this purpose the sources chosen should be such that as little heat as possible will be generated by them. Small incandescent electric lights are best for this purpose. Refinements of this kind cannot always be used, of course, but the prime requisite with reference to the avoidance of temperature errors is that all operations—filling the flasks and tubes, reading the solutions, controlling the instrument with standard quartz plates, etc.—should be done at one and the same temperature, and that this temperature be a constant one, that is, not varying greatly at different hours of the day. For example, the room should not be allowed to become cold at night, so that it is at low temperature in the morning when work is begun, and then rapidly heated up during the day. The polariscope should not be exposed to the direct rays of the sun during part of the day, and should not be near artificial sources of heat, such as steam boilers, furnaces, flues, etc.

The tables upon which the instruments stand should be level.

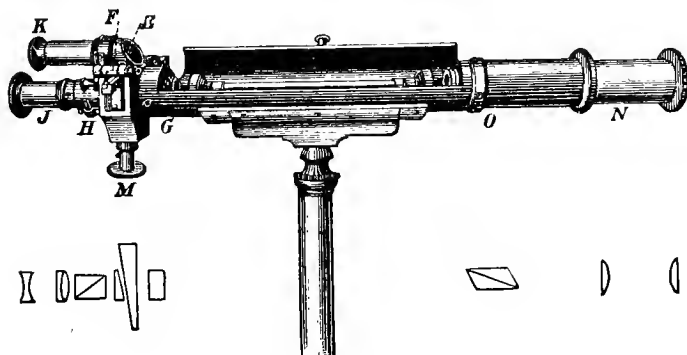


FIG. 1.—Haensch's half-shadow saccharimeter.

METHODS OF MANIPULATION

The methods of manipulation used in the polarisation of sugar are of prime importance. They consist in weighing out the sugar, dissolving it, clarifying the solution, making it up to standard volume, filtering, filling the observation tube, regulating the illumination, and making the polariscopic reading.

The proper conduct of these processes, in connection with the use of accurately graduated apparatus, is the only surety against the numerous sources of error which may be encountered. Different sugars require different treatment in clarification, and much must necessarily be left to the judgment and experience of the operator.

The following directions are based upon various official pro-

cedures, such as the one used in the United States custom-houses, the method prescribed by the German Government, etc. They embody also the results of recent research in regard to sources of error in the polarimetric estimation of sugar.

DIRECTIONS FOR THE POLARISATION OF SUGAR

Description of Instrument and Manner of Using

The instrument employed is known as the half-shadow apparatus of Schmidt & Haensch. It is shown in Fig. 1.

The tube N contains the illuminating system of lenses, and is placed next to the lamp; the polarising prism is at O and the analysing prism at H. The quartz wedge compensating system is contained in the portions of the tube marked F, E, G, and is controlled by the milled head M. The tube J carries a small telescope, through which the field of the instrument is viewed, and just above is the reading tube K, which is provided with a mirror and magnifying lens for reading the scale.

The tube containing the sugar solution is shown in position in the trough between the two ends of the instrument. In using the instrument, the lamp is placed at a distance of at least 200 millimetres from the end; the observer seats himself at the opposite end in such a manner as to bring his eye in line with the tube J. The telescope is moved in or out until the proper focus is secured, so as to give a clearly defined image, when the field of the instrument will appear as a round, luminous disc, divided into two halves by a vertical line passing through the centre, and darker on one half of the disc than on the other. If the observer, still looking through the telescope, will now grasp the milled head M and rotate it, first one way and then the other, he will find that the appearance of the field changes, and at a certain point the dark half becomes light and the light half dark. By rotating the milled head delicately backwards and forwards over this point he will be able to find the exact position of the quartz operated by it, in which the field is natural or of the same intensity of light on both halves.

The three different appearances presented by the field are best shown in the following diagram—

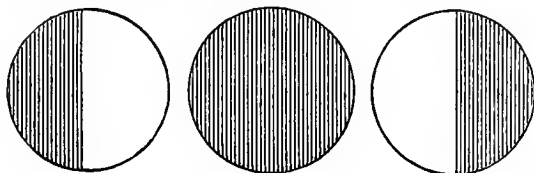


FIG. 2.

With the milled head set at the point which gives the appearance of the middle disc as shown above, the eye of the observer is raised to the reading tube K, and the position of the scale is noted. It will be seen that the scale proper is attached to the quartz wedge, which is moved by the milled head, and attached to the other quartz is a small scale called a vernier, which is fixed, serving for the exact determination of the movable scale with reference to it. On each side of the zero line of the vernier a space corresponding to nine divisions of the movable scale is divided into ten equal parts. By this device the fractional part of a degree indicated by the position of the zero line is ascertained in tenths; it is only necessary to count from zero until a line is found which makes a continuous line with one on the movable scale.

With the neutral field, as indicated above, the zero of the movable scale should correspond closely with the zero of the vernier, unless the zero point is out of adjustment.

If the observer desires to secure an exact adjustment of the zero of the scale, or in any case if the latter deviates more than one half of a degree, the zero lines are made to coincide by moving the milled head and securing a neutral field at this point by means of the small key which comes with the instrument, and which fits a small nipple on the left side of F, the fixed quartz wedge of the compensating system. This nipple must not be confounded with a similar nipple on the right-hand side of the analysing prism H, which it fits as well, but which must never be touched, as the adjustment of the instrument would be seriously disturbed by moving it. With the key on the proper nipple, it is turned one way or the other until the field is neutral. Unless the deviation of the zero be greater than $0^{\circ}5$ it will not be necessary to use the key, but only to note the amount of the deviation; and for this purpose the observer must not be content with a single setting, but must perform the operation five or six times and take the mean of these different readings. If one or more of the readings show a deviation of more than $0^{\circ}3$ from the general average, they should be regarded as incorrect. Between each reading the eye should be allowed ten to twenty seconds of rest.

The "setting" of the zero having been performed as above, the determination of the accurate adjustment of the instrument by means of the control quartz plates is proceeded with. Three such plates will be furnished with each polariscope, which have sugar values respectively approximating 80° , 90° , and 100° . These values vary with the temperature, and tables are furnished with them giving their exact value at different temperatures from 10° to 35° C.

One of these plates is placed in the instrument and the field observed. The milled head is turned to the right until the exact point of neutrality is re-established, just described above in setting the zero. The scale is read, the observation repeated, the reading

again taken, and so on until five or six readings have been made. The average is taken, readings being rejected which show a divergence of more than 0.3, and the result corrected for the deviation of the zero point, if any was found, the deviation being added if it was to the left, and subtracted if to the right. If the adjustment of the instrument be correct, the result should be the value of the control plate used as ascertained from the table for the temperature of 20° C. Each of the three plates is read in the instrument in this way. A variation of 0.3 from the established values may be allowed for errors of observation, but in the hands of a careful observer a deviation greater than this with one of the three plates, after a careful setting of the zero, shows that the instrument is not accurately adjusted.

The complete verification of the accurate adjustment of the polariscope by means of three control plates as given above should be employed wherever it is set up for the first time by those about to use it, whenever it has sustained any serious shock or injury, and whenever it has been transported from one place to another. It should also be done at least once a week when the instrument is in constant use.

After the complete verification has been performed as described, further checking of the instrument is done by means of one control plate alone, the one approximating 90°, and the setting of the zero point is dispensed with, the indication of the scale for sugar solutions being corrected by the amount of deviation shown in the reading of the 90° control plate from its established value as ascertained from the table at the temperature of the room.

For example, a sugar solution polarises 80°·5; the control plate just before had given a polarisation of 91°·4, the temperature of the room during both observations being 25° C. According to the table, the value of the control plate at 25° C. is 91°·7; the reading is therefore 0°·3 too low, and 0°·3 is added to the reading of the sugar solution, making the corrected 80°·8. The temperature of the room should be ascertained from a standardised thermometer placed close to the instrument, and in such a position as to be subject to the same conditions.

For the daily control of an optical saccharimeter there is perhaps nothing superior, says Wiley, to a first-class quartz plate, the value of which has been tested against pure sugar. The danger of using a quartz plate at its marked value is very great indeed. The quartz plate also has different values according to the position in which it is placed in the instrument, so that its value should be fixed once for all by comparison against a pure sugar solution. A control tube may also be used, which can be made of variable length by ratchet and scale over a carefully graduated vernier, using pure sugar and solution for charging the tubes. An optical saccharimeter should

thus be carefully graduated at each 5° from zero to 100° , and any inaccuracy in the scale noted and allowed for in actual practice.

PORTABLE SACCHARIMETER

The instrument by Carl Zeiss of Jena (Fig. 3) consists of a glass tube *A* of exactly 200 millimetres length, closed by parallel plates of glass *s, s*, at both ends, which is inserted into a brass tube *B*. This brass tube carries a divided circle *c*, divided into half-degrees, and upon this circle a shorter tube *D* rotates and contains a Nicol prism *a* (the analyser). The rotation of *D* is measured on the divided circle by means of an index pointer *d*. The other end of *B* bears, by means of an eccentric pivot, a short tube *E* with another Nicol prism *p* (the polariser), and a double plate of rock crystal *g*.

If *E* be turned aside round the pivot the tube *B* is opened and the glass tube *A* can be slipped out, whilst *A* is held in its place when *E* is in its centric position.

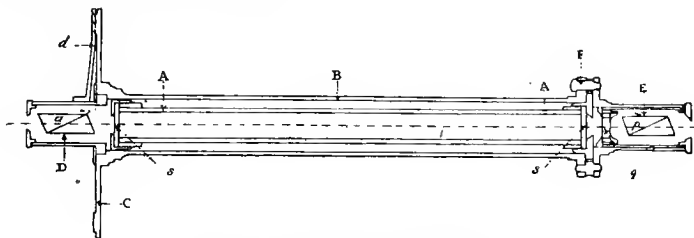


FIG. 3.—Zeiss's portable saccharimeter.

To examine the rotatory power of a solution, open the tube *B* by turning aside *E* and slip out the glass tube *A*; unscrew one of the plates *s* (the tube and both plates *s* having been previously cleaned), and fill the tube in an upright position with the liquid to be examined; then shut it by screwing on the glass plate, taking care that no air-bubbles remain within. Insert the glass tube into *B* and fix it by turning back *E*. Now direct the instrument to the bright sky or other source of light, while looking through the opening at the end of *D*. The clear surface of the rock-crystal plate *g* will be seen as a circle with two halves of distinct colours; turn the rotating eye-piece *D* until both semicircles acquire the same colour as near as possible, namely a reddish violet. This obtained, read off the position of the index pointer *d* on the divided circle. Provided the instrument be properly adjusted, the reading indicates the rotatory power of the solution for a layer of 200 millimetres in degrees and fractions of a degree. In order to test the instrument, fill the glass tube with distilled water, turn the index *d* to the zero point of the circle. If the adjustment be correct, the two halves of the plate *g* will appear of

the same colour. If it is not adjusted the tube E must be turned round its axis within the external tube in which it is inserted, which can be done after loosening a small screw by which it is fixed.

In a tube 200 millimetres long a solution of 1 gramme of cane sugar in 100 c.c. of water rotates $1^{\circ}33$. Under the same circumstances invert sugar rotates $-1^{\circ}06$; 1° indicates, therefore, in the case of cane sugar, $0.75 (= \frac{3}{4})$ per cent.; and, in the case of invert sugar, 0.94 per cent., and any number of degrees read off on the divided circle proportionately more.

Regarding other substances which may be tested by the instrument, the necessary data must be obtained by experiments or by reference to some work on the subject.

IV.—CORRECTIONS OF POLARISATIONS AT DIFFERENT TEMPERATURES (MATEGEZECK).

° C.	Instead of 100.
15	100.05
16	100.03
17	100.01
17½	100.00
18	99.99
19	99.96
20	99.94
21	99.90
22	99.89
23	99.87
24	99.85
25	99.82

Influence of temperature on the specific rotary power of cane sugar.—The specific rotary power of cane sugar is generally acknowledged to be independent of temperature. If, however, a solution of sugar be made up to the standard volume of 100° C. at a certain temperature, and then polarised at *different* temperatures, it will show either an apparent increase or decrease in rotation power as the temperature is lowered or raised (Table IV.). This is simply due to the fact of the contraction of the solvent, thus admitting a larger number of sugar molecules in a given length of the column at a low than at a high temperature.

Waetze found that the polarisation expressed in degrees of cane sugar in a normal solution (26.048 grammes in 100 c.c.) changes about 0.1 of a degree for each difference of 2° C. in temperature. A solution polarised at 14° C. gave a reading of $95^{\circ}6$. The same solution at 34° C. gave a polarisation of $94^{\circ}7$, or $0^{\circ}9$ for a difference of 20° C. In another experiment a difference of 0.1 of a polarisation degree was

shown for a difference of 24° C. On changing the temperature back to the original, the original polarisation was obtained. A solution polarised at 17° C. gave 95°·1, but cooled to 10° C. it showed 95°·5; when reheated to 17° C. it again showed 95°·1. The above results show the importance of polarising the solution at the temperature at which it is made. For this reason the solutions should all be made at a given temperature in a place near that where the polarisations are made, and the temperature should not be allowed to change from the time the solutions are made until the polarisations are complete.

Wiley points out that most sugar flasks are graduated either at 15° or 17°·5, and that the mean temperature in the U.S.A. is much higher than this, and that it will probably be well to have all U.S.A. flasks graduated at about 22°. Since, however, the volume of a flask will change very little within the limits given, it is more important that the polarisations be made at the same temperature at which the solution is made up, than that all the flasks should be graduated at a given temperature.

V.—ROTARY POWER OF CANE SUGAR IN DIFFERENT DEGREES OF CONCENTRATION, ACCORDING TO THE FORMULÆ OF TOLLENS, SCHMITZ, AND LANDOLT.

C.	[α]D.	C.	[α]D.
4	66·63	17	66·51
5	66·62	18	66·50
6	66·61	19	66·49
7	66·60	20	66·48
8	66·59	21	66·47
9	66·58	22	66·46
10	66·57	23	66·45
11	66·57	24	66·44
12	66·56	25	66·43
13	66·55	26	66·42
14	66·54	27	66·41
15	66·53	28	66·40
16	66·52		

[TABLE VI.]

VI.—ROTARY POWER OF SUGARS.

Substance.	° C.	Limits of Concentration.	Rotary Power $[\alpha]_D$.	Observers.
Cane sugar, $C_{12}H_{22}O_{11}$ the solution at 17°·5 compared with water at 4°	20	$p = 4 - 18$	$+ 66\cdot810 - 0\cdot015553p - 0\cdot000052462p^2$	Tollens.
„	20	$p = 18 - 69$	$+ 66\cdot386 + 0\cdot015035p - 0\cdot0003986p^2$	„
„	20	$p = 5 - 18$	$+ 66\cdot727 - 0\cdot015534p + 0\cdot000052396p^2$	„
„	20	$p = 18 - 69$	$+ 66\cdot303 + 0\cdot015016p - 0\cdot0003981p^2$	„
„	20	$p = 35 - 98$	$+ 64\cdot156 + 0\cdot05196p + 0\cdot00028052p^2$	Schmitz.
„	20	$c = 4 - 28$	$+ 66\cdot67 - 0\cdot00955c$	Landolt.
„	20	$p = 0\cdot5 - 1\cdot2$	$+ 66\cdot96 - 4\cdot6896p + 1\cdot8615p^2$	Nasini and Vilevechia,
„	20	$p = 3 - 65$	$+ 66\cdot438 + 0\cdot010312p - 0\cdot00035449p^2$	Prübram.
„	20	$p = 0\cdot2 - 4$	$+ 64\cdot262 - 0\cdot6063p + 2\cdot346p^2$	Hesse.
„	15	$c = 0 - 10$	$+ 68\cdot65 - 0\cdot828c + 0\cdot115415c^2[\alpha]_D = 75\cdot08$ for $[\alpha]_D = 66\cdot5$	„
„	15	$c = 5$	$+ 66\cdot7$ alcoholic solution	Muntz.
„	„	$c = 10$	$+ 67$	„
„	„	„	$+ 64\cdot9$	„
„	„	„	$+ 61\cdot3$	„
„	„	„	$+ 46\cdot9$	„
„	„	„	$+ 51\cdot8$	„
„	„	„	$+ 66$	„
„	15	$c = 5$	$0\cdot170$	Hesse.
„	„	$c = 5\cdot4$	$0\cdot140$	Pellet.
„	„	$5\cdot4$	„	„
„	20	$p = 0 - 100$	$+ 52\cdot50 + 0\cdot018796p + 0\cdot00051683p^2$	Tollens.
„	20	$p = 0 - 100$	$+ 47\cdot73 + 0\cdot015534p + 0\cdot0003883p^2$	„
„	15	$c = 2\cdot8$	$+ 51\cdot78$	Hesse.
„	15	$c = 3$	$+ 51\cdot67$	„
„	15	$c = 2\cdot5$	$+ 51\cdot80$	„
„	15	$c = 2\cdot0$	$+ 49\cdot25$	„
Cane sugar + 0·409g = $\frac{1}{2}$ mol. $C_{12}H_{22}O_{11}$	„	„	„	„
„ + 0·818g = $\frac{1}{2}$ mol. CaO	„	„	„	„
„ + 1·637g = 1 mol. CaO	„	„	„	„
„ + 3·274g = 2 mol. CaO	„	„	„	„
„ + 1 mol. Na_2O	„	„	„	„
„ + 1g KHO in 100 c. c.	„	„	„	„
„ + 1g NaHO in 100 c. c.	„	„	„	„
Dextrose anhydrous, $C_6H_{12}O_6$	„	„	„	„
„ $C_6H_{12}O_6 \cdot H_2O$	„	„	„	„
„ Grape sugar, $C_6H_{12}O_6$	„	„	„	„
„ Starch sugar, $C_6H_{12}O_6$	„	„	„	„
„ Salicin sugar, $C_6H_{12}O_6$	„	„	„	„
„ Amygdalin sugar, $C_6H_{12}O_6 + H_2O$	„	„	„	„

Glucose, $C_6H_{12}O_6 + H_2O$	20	$\rho = 8 - 21$	$+17.925 + 0.015534\rho + 0.000388\rho^2$	Tollens,
" $C_6H_{12}O_6$	20	$\rho = 7 - 83$	$+52.718 + 0.017037\rho + 0.0004271\rho^2$	"
" $+ 0.985 C_6H_{12}O_6$	15	$c = 6.9$	$+ [a]_j = +33.3$	Jodin.
	14		$+ [a]_j = -106$	Dubrunfant.
	52		$+ [a]_j = -79.5$	"
	90		$+ [a]_j = -53$	Neubauer.
	14	12.8	$+ [a]_j = -100$	Jodin.
Levulose, $C_6H_{12}O_6$	0-40	$c = 0 - 40$	$+ [a]_j = -104$	Junfleisch and
			$-100.3 - 0.108c + 0.56t$	Gronbert.
	20	$c = 1 - 14$	$+ 20.07 - 0.041c$	Hammersmidt.
	14		$[a]_j = -26.65$	Dubrunfant.
	52		$[a]_j = -13.32$	"
	90		$[a]_j = 0$	"
Invert sugar, $C_6H_{12}O_6$	0	$c = 17 - 21$	$-27.9[a]_j D = -(27.9 - 0.32t)$	Tuchsmid.
Lactose, $C_{12}H_{22}O_{11} + H_2O$	20	$\rho = 0 - 36$	$+52.47$ constant	Berthelot.
" $C_{12}H_{22}O_{11}$	15	$c = 2 - 12$	$+54.54 - 0.547\rho + 0.05475c^2$	Hesse.
Maltose, $C_{12}H_{22}O_{11}$	20	$\rho = 0 - 36$	$+137.04$	Ost.
Galactose, $C_6H_{12}O_6$	10-30	$\rho = 5 - 35$	$+83.883 + 0.0785\rho - 0.209t$	Meissst.
Arabinose, $C_6H_{12}O_6$	20	$\rho = 10$	$+105.5$ constant	Von Lippmann, etc.
Arabinic acid, $C_6H_{10}O_5$		$c = 5$	$[a]_j = +37.3 + 46.1 - 28.8 - 29.2 - 30$	Scheibler.
Raffinose, $C_{18}H_{32}O_{16} + 5 Aq$	20	$\rho = 0 - 10$	$+104.5$ constant	Loiseau, etc.
Tetraacetylactose, $C_{12}H_{18}(C_2H_3O)_4O_{11}$		$c = 7.46$	$+50.1$	Schitzenberger.
Mycose, $C_{12}H_{22}O_{11} + 2 H_2O$	15	$c = 10.03$	$[a]_j = +173.2$	Mitscherlich.
Trehalose, $C_{12}H_{22}O_{11} + 2 H_2O$	25	$c = 8.4$ to 14.8	$[a]_j = +199$	Berthelot.
Melitose, $C_{12}H_{22}O_{11} + 3 H_2O$	20	$c = 17.27$	$[a]_j = +88$	"
Melzitose, $C_{12}H_{22}O_{11} + H_2O$		$c = 18.6$	$[a]_j = +94.1$ (anhydrous)	"
Sorbin, $C_6H_{12}O_6$		$c = 23.9$	$[a]_j = -46.9$	"
Mannit, $C_6H_{14}O_5$		$c = 15$	$[a]_j = -0.03$	Pasteur.
Nitromannit, $C_6H_8(O.NO)_2$		$c = 7.5$	$+40$ alcoholic solution	Krusenmann.
Isodulcit, $C_6H_{12}O_6 + H_2O$		$c = 10.2$	$[a]_j = 47.6$	Hlasiwetz.
Quercit, $C_6H_{12}O_6$	16	$c = 1$ to 10	$+24.3$	Prunier.

VOLUMETRIC ANALYSIS OF SUGARS

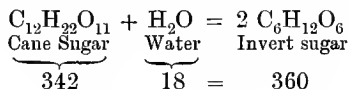
It has been claimed for this method that it is the most expeditious, the easiest, and, when well performed, the most exact process of sugar analysis.

The principle of the method depends on the three following facts :—

1. *Cane sugar* has no action on the special copper solution known as Bareswill's liquor (Fehling's solution).

2. All *glucoses*, on the contrary, reduce this solution and precipitate its copper as cuprous oxide.

3. Cane sugar, which has no action on the boiling solution of copper, fixes water under the action of heat and mineral acids (hydrochloric and sulphuric), and is transformed in equal proportion into two new sugars, glucose and levulose—a mixture which has been termed *invert sugar*—



REAGENTS

1. *Sulphuric acid for inversion*.—100 grammes of sulphuric acid are run in a thin stream into 200 grammes of distilled water with constant stirring. The mixture becomes very hot. When it is cold it is preserved in a well-stoppered bottle.

2. *Normal sugar solution ½ per cent.*—1 gramme of pure dry sugar candy is dissolved in 50 c.c. of distilled water, to which about 2 c.c. of sulphuric acid are added to invert the sugar by boiling for a quarter of an hour. The operation is performed in a graduated flask of 200 c.c. capacity. When the solution is cold it is made up to 200 c.c., and closed with the thumb, and the flask turned repeatedly upside down so as to thoroughly mix the solution and render it uniform throughout.

This solution therefore contains ½ per cent. of cane sugar in the form of invert sugar.

3. *Copper solution (Violette's) for the estimation proper*.—Particular care must be devoted to the preparation of this liquid.

Solution 1.

36.46 grammes of sulphate of copper are dissolved in 140 c.c. of distilled water.

Solution 2.

200 grammes of the neutral tartrate of potash and soda are dissolved in 600 c.c. of caustic soda of 22° B. (sp. gr. 1.2).

The second solution is run into a 1-litre flask, and the first solution added in small portions at a time; finally, the bulk is made up to a litre with distilled water, which is first used for rinsing the dishes

containing the sulphate of copper and the double tartrate of potash and soda.

If the sulphate of copper was pure enough 10 c.c. of this liquid would require for its reduction 0.050 gramme of cane sugar, which would correspond to 0.0526 gramme of glucose. But before taking this for granted it is necessary to verify it.

By means of a 10 c.c. pipette 10 c.c. of the copper solution are run into a small Bohemian glass flask of 100 c.c. capacity, or, better still, into a test tube containing a few fragments of pumice to prevent bumping on boiling the liquid.

On the other hand, the Mohr's burette is filled to the upper division with the standard solution of sugar ($\frac{1}{2}$ per cent.).

This being done, the copper solution is rapidly boiled, and the sugar solution run in drop by drop. After each addition the liquid is boiled, whilst the flask is shaken. The liquid at first loses its transparency, becomes yellow and then red, and finally becomes completely decolorised. As this moment draws nigh after each addition of the standard sugar solution, the flask is placed for an instant on white paper so as to seize the precise moment when the liquid has become perfectly colourless. A final drop of sugar solution is then added to make sure that no precipitate is formed, and when that result is obtained the operation is finished. The volume of liquid is then read off from the scale on the burette. Suppose that the number of divisions were equal to 10 c.c., then

$$\begin{array}{cccc} \text{C.c.} & \text{C.c.} & \text{Gramme} & \text{Gramme} \\ 10 & : & 100 & :: 0.50 & : & x = 0.5 \end{array}$$

The strength of the standard cupro-potassic solution is therefore exact. The formula adopted is that of Violette, which is to be preferred to Fehling's or Mohr's, as the solution keeps better.

The real titrations are simply repetitions of the first experiment.

Volumetric estimation of sugar.—The complete analysis of a sugar ought to give—

1. The percentages of glucoses.
2. The percentage of cane sugar.
3. Moisture and impurities.

These results are obtained by two consecutive experiments.

Total sugar.—1 gramme of the sugar is dissolved in about 50 c.c. of distilled water, to which 2 to 3 c.c. of acid are added for inversion. The solution is boiled for a quarter of an hour over a spirit lamp or bunsen burner. The heat is then withdrawn and the solution allowed to cool, and then filtered into a 200 c.c. flask. The first flask in which the solution was dissolved is very carefully washed, and the wash water run through the filter so as to remove all trace of sugar. The filtrate is then made up to 200 c.c. with distilled water. The mouth of the flask is then closed with the thumb of the right hand,

and the flask turned repeatedly upside down so as to obtain a perfectly homogeneous solution.

The Mohr's burette having been carefully washed with distilled water and dried by means of a tube enveloped in filter paper, which is caused to glide along its inside surface, is filled with the standard sugar solution, and a new test is made similar to the first.

Suppose 10.8 c.c. of the sugar solution were required to reduce 10 c.c. of the cupro-potassic solution, we then get—

$$\begin{array}{cccc} \text{C.c.} & \text{C.c.} & \text{Gramme} & \text{Gramme} \\ 10.8 & : 200 & :: 0.050 & : x = 0.9259 \end{array}$$

1 gramme of the sample therefore contained 0.9259 gramme of sugar, say 92.59 per cent. Table VII. obviates calculation.

It now remains to be seen whether the sample contained any glucose. That point is determined as follows:—

Estimation of glucose.—A fresh amount of sugar—10 grammes—is again weighed out and dissolved in about 50 c.c. of water, and filtered as before into the graduated flask, and brought by the addition of the wash-water to 100 c.c. only. The flask is turned upside down repeatedly to mix the whole of the layers of liquid. The Mohr's burette having been washed and dried is filled with the sugar solution, and a fresh titration with the cupro-potassic solution is performed.

Suppose that 16.1 c.c. of sugar solution were required to reduce 10 c.c. of cupro-potassic solution, we get—

$$\begin{array}{cccc} \text{C.c.} & \text{C.c.} & \text{Gramme} & \text{Gramme} \\ 16.1 & : 100 & :: 0.050 & : x = 0.310 \end{array}$$

Now 0.310 gramme for 10 grammes of sugar = 3.10 per cent. of glucose expressed as cane sugar.

Result.

Total sugar	92.59 per cent.
Glucose	3.10 ,,
					89.49
Difference	,,

The difference gives the amount of cane sugar.

To arrive definitely at the exact composition of the sugar analysed, all that has to be done is to convert the result of the second experiment, in which the glucose is expressed as cane sugar, into real glucose, $C_6H_{12}O_6$. This is done by multiplying the result by 1.052—
 $3.10 \times 1.052 = 3.26$.

COMPOSITION OF THE SUGAR ANALYSED

Cane sugar	89.49 per cent.
Glucose	3.26 ,,
					92.75
Moisture and impurities by difference					7.25 ,,
					100.00
					,,

VII.—PROPORTION OF CANE SUGAR CONTAINED IN IMPURE SUGARS OR SACCHARINE JUICES.

I. $\frac{1000}{V}p$ 100. II. $\frac{50}{\sqrt{V}}p$ 100. (Ville).

I. Rich Sugars.		II. Saccharine Juices.		Recapitulation of Principles.
Volume of Sugar Solution used.	Cane Sugar per cent.	Volume of Sugar Solution used.	Cane Sugar per cent.	
c.c.		c.c.		
10	100·0	1	50	
10·1	99·01	1½	33·33	I. 10 molecules of <i>cane sugar</i> produce 20 molecules of glucose. (See equation, p. 26.)
10·2	98·04	2	25	II. 1 part of <i>cane sugar</i> corresponds to 1·052 parts of glucose.
10·3	97·09	2½	20	III. <i>Cane sugar</i> has no action on Fehling's solution.
10·4	96·15	3	16·07	IV. <i>Glucoses</i> precipitate Fehling's solution as cuprous oxide.
10·5	95·24	3½	14·29	V. One equivalent of <i>glucose</i> (C ₆ H ₁₂ O ₆) precipitates 10 equivalents of sulphate of copper (CuSO ₄ , 5 H ₂ O).
10·6	94·34	4	12·50	180 grammes of <i>glucose</i> correspond to 1246·8 of sulphate of copper, and 5 grammes of <i>glucose</i> correspond to 34·633 grammes of sulphate of copper.
10·7	93·46	4½	11·11	
10·8	92·59	5	10	
10·9	91·74	5½	9·09	
11	90·91	6	8·33	
11·1	90·09	6½	7·69	
11·2	89·29	7	7·14	
11·3	88·50	7½	6·67	
11·4	87·72	8	6·25	
11·5	86·96	8½	5·88	
11·6	86·21	9	5·56	
11·7	85·47	9½	5·26	
11·8	84·75	10	5	
11·9	84·03	10½	4·76	
12·0	83·33	11	4·55	
12·1	82·64	12	4·17	
12·2	81·97	13	3·85	
12·3	81·30	14	3·57	
12·4	80·64	15	3·33	
12·5	80	16	3·12	
12·6	79·36	17	2·94	
12·7	78·74	18	2·78	
12·8	78·12	19	2·63	
12·9	77·52	20	2·50	
13	76·92	22½	2·22	
13·1	76·34	25	2	
13·2	75·76	27½	1·82	
13·3	75·19	30	1·67	
13·4	74·64	32½	1·54	
13·5	74·07	35	1·43	
13·6	73·53	37½	1·33	
13·7	72·99	40	1·25	
13·8	72·46	42½	1·18	
13·9	71·94	45	1·11	
14	71·43	47½	1·05	
...	...	50	1	

Table I. refers to a solution of 1 gramme of cane sugar in 200 c.c. of distilled water.

VIII.—PHENOMENA OBSERVED IN TITRATING GLUCOSE WITH COPPER SOLUTION (VIOLETTE).

Phases of the Operation.	Fundamental Characteristics.		Secondary Characteristics.	
	Precipitation on the Surface of the Hot Clear Liquid.	Coloration on the Surface of the Hot Clear Liquid.	Colour of the Mass in Full Boil.	Colour of the Liquid Floating on the Deposit.
Commencement.	Yellow precipitate, then orange, then red.	Same shades as that of the whole liquid as given in the fifth column.	Deep blue violet.	Deep blue.
Towards the middle.	Orange red precipitate very perceptible.		Reddish violet.	Light blue.
Towards the end.	Orange red precipitate.		Brick red.	Very pale blue.
End.	Slight cloudiness.		Decided red.	''
	Final cloudiness scarcely perceptible.		Bright red.	''
Beyond the finish.	No precipitate.	Amber transparent zone. Transparent zone, deeper coloured. Transparent zone colour of gamboge. Deep brown transparent zone.	Beautiful bright red.	Colourless.
''	''		''	''
''	''		''	''
''	''		Bright red, a little yellow.	Straw yellow.
''	''		Red, slightly brown.	Amber.
				Deeper.
				Gamboge yellow.

Note.—The froth of the liquid on boiling, at first blue, becomes colourless at the end of the operation, then yellow and brownish yellow when the precipitation is gone beyond. The liquid then gives off a burnt-sugar smell.

IX.—ESTIMATION OF GLUCOSE IN RAW SUGAR.

Solution of 16·29 Grammes Sugar in 100 c.c.				Sugar Solution treated with 10 per cent. of Carbonate of Soda.			
Working on 1 c.c. of Fehling's Solution.		Working on $\frac{1}{2}$ c.c. of Fehling's Solution.		Working on 1 c.c. of Fehling's Solution.		Working on $\frac{1}{2}$ c.c. of Fehling's Solution.	
c.c. Sugar Solution.	Glucose per cent.	c.c. Sugar Solution.	Glucose per cent.	c.c. Sugar Solution.	Glucose per cent.	c.c. Sugar Solution.	Glucose per cent.
5·1	0·60	2·6	0·60	5·7	0·60	2·9	0·60
5·6	0·55	2·8	0·55	6·2	0·55	3·1	0·55
6·2	0·50	3·1	0·50	6·8	0·50	3·4	0·50
6·9	0·45	3·5	0·45	7·5	0·45	3·8	0·45
7·7	0·40	3·9	0·40	8·5	0·40	4·3	0·40
8·8	0·35	4·4	0·35	9·7	0·35	4·9	0·35
10·3	0·30	5·2	0·30	11·3	0·30	5·7	0·30
12·4	0·25	6·2	0·25	13·6	0·25	6·8	0·25
15·5	0·20	7·8	0·20	17·0	0·20	8·5	0·20
20·6	0·15	10·3	0·15	22·7	0·15	11·4	0·15
30·9	0·10	15·5	0·10	34·0	0·10	17·0	0·10
61·8	0·05	30·9	0·05	68·0	0·05	34·0	0·05

X.—VOLUMETRIC ESTIMATION OF GLUCOSE AND LEVULOSE, LACTOSE AND MALTOSE.

(Table for use with solutions containing 0·1 to 0·5 per cent. of reducing sugars, —titrated with 10 c.c. Fehling's solution,—the results being expressed in grammes per litre or in lbs. per 100 gallons.)

C.c. Sugar Solution.	Glucose and Levulose.	Lactose (anhydr.).	Maltose (anhydr.).	C.c. Sugar Solution.	Glucose and Levulose.	Lactose (anhydr.).	Maltose (anhydr.).
10	5·000 Diff. ·454	6·350	7·500	17	2·941 Diff. ·163	3·744	4·412
11	4·546 ·379	5·773	6·818	18	2·778 ·146	3·528	4·167
12	4·167 ·321	5·292	6·250	19	2·632 ·132	3·342	3·947
13	3·846 ·275	4·884	5·770	20	2·500 ·119	3·175	3·750
14	3·571 ·237	4·536	5·357	21	2·381 ·108	3·024	3·571
15	3·334 ·209	4·233	5·000	22	2·273 ·099	2·886	3·409
16	3·125 ·184	3·969	4·687	23	2·174 ·091	2·761	3·261

[TABLE CONTINUED.]

X.—VOLUMETRIC ESTIMATION OF GLUCOSE, ETC.—*continued.*

C.c. Sugar Solution.	Glucose and Levulose.	Lactose (anhydr.).	Maltose (anhydr.).	C.c. Sugar Solution.	Glucose and Levulose.	Lactose (anhydr.).	Maltose (anhydr.).
24	2·083	2·643	3·125	32	1·563	1·984	2·344
	Diff. ·083				Diff. ·048		
25	2·000	2·540	3·000	33	1·515	1·924	2·272
	·077				·044		
26	1·923	2·442	2·884	34	1·471	1·868	2·206
	·071				·042		
27	1·852	2·354	2·778	35	1·429	1·814	2·143
	·066				·040		
28	1·786	2·268	2·679	36	1·389	1·764	2·083
	·062				·038		
29	1·724	2·189	2·586	37	1·351	1·717	2·027
	·057				·035		
30	1·667	2·117	2·500	38	1·316	1·671	1·973
	·054				·034		
31	1·613	2·049	2·419	39	1·282	1·628	1·923
	·050				·032		
				40	1·250	1·588	1·875

Politis's rapid method for the estimation of sugar with $\frac{N}{10}$ copper-alkali solution.—The determination of sugar with Fehling's solution would be accurate and rapid enough if the end of the reaction—that is, the complete decolorisation of the alkaline copper solution—was easy to determine. The suspended copper suboxide makes this very difficult. To avoid this inconvenience and to make the determination more accurate and rapid, the author uses a definite amount of sugar solution, an excess of $\frac{N}{10}$ copper solution, and estimates the excess according to De Haen's method, with potassium iodide and sodium hyposulphite. The following solutions are required:—

(1) $\frac{N}{10}$ copper solution consisting of 24·95 grammes pure copper sulphate, 140 grammes sodium potassium tartrate, 25 grammes pure sodium hydrate, and sufficient water to make 1 litre.

(2) $\frac{N}{10}$ solution of sodium hyposulphite, 24·8 grammes per litre.

(3) $\frac{N}{10}$ iodine solution, 12·7 grammes per litre. The three solutions must correspond exactly, and 1 c.c. of the copper solution is equivalent to 0·0036 gramme glucose.

The estimation is made as follows:—

50 c.c. of the copper solution are boiled in a porcelain dish and 10 c.c. of a sugar solution added, which contains about 1 gramme glucose in 1000. The whole is heated for five minutes, made up to 100 c.c., and exactly 50 c.c. filtered into a beaker. The blue liquid is made slightly acid, a slight excess of potassium iodide and some starch solution added, and the iodine estimated by means of the $\frac{N}{10}$ sodium hyposulphite solution. This iodine corresponds to the un-reduced copper in the liquid, and the copper required by the glucose can be calculated by difference. The number of c.c. $\frac{N}{10}$ $\text{Na}_2\text{S}_2\text{O}_3$ solution required is deducted from 25, and the result will be the number of c.c. of copper solution which have been reduced by 5 c.c. of the sugar solution.

Example.—50 c.c. $\frac{N}{10}$ copper solution were boiled with 10 c.c. of diabetic urine which had been diluted to 5 times its volume, and the estimation completed as above. 11 c.c. of $\frac{N}{10}$ $\text{Na}_2\text{S}_2\text{O}_3$ were required to completely decolorise the liquid; $25 - 11 = 14$ c.c. of copper solution which had been reduced by one-half of the sugar solution, what is 5 c.c. of diluted or 1 c.c. of the original urine? As 1 c.c. of copper solution = 0.0036 gramme glucose, 1 c.c. of urine contained 0.0504, or 1 litre 50.4 grammes glucose.

Soldaini's solution.—A solution of copper carbonate in potassium carbonate was proposed as a substitute for Fehling's solution by Arturo Soldaini.

Modifications of Soldaini's method.—1. *Striegler's modifications.*—Striegler made a number of experiments to find means by which Soldaini's reagent could be prepared with a definite copper content. He first examined Bodenbender and Scheller's method of preparing this reagent, and then elaborated a method by which a constant solution can be prepared. He followed Degener, Schweitzer, and Preuss's instructions so as to get accurate results. Scheller's and Preuss's researches are summarised on pp. 36 and 37. Analyses of various portions of the reagent prepared by the same method show—

(1) That almost one-half the potassium carbonate used has been decomposed with the evolution of CO_2 .

(2) That the amount of copper dissolved varies considerably between 131.1 and 139.7 milligrammes copper in 100 c.c.; with regard to Preuss's figures, even from 104 to 187 milligrammes copper in 100 c.c.

(3) That the quantity of copper dissolved is very low as compared with the quantity used. Used = 0.727 gramme copper dissolved at the most = 0.1597 gramme copper in 100 c.c.

Further experiments proved that on dissolving the potassium carbonate in hot water a large amount is decomposed, and that in preparing the reagent more is used than can remain in solution at the ordinary temperature. It is therefore necessary, after preparing the reagent, to decompose bicarbonate at least in part, so as to prevent any excess from crystallising out. This can easily be accomplished by boiling.

As the result of further experiments, Striegler prepared a reagent of definite composition, as follows:—

An amount of copper sulphate corresponding to 1.025 gramme copper in 100 c.c. was accurately weighed out, dissolved in cold water, made alkaline with soda lye, and the precipitate washed well. The usual amount of potassium bicarbonate was then added, and the whole heated at 40° to 45° C. until the bicarbonate had dissolved, and then at 60° until complete solution of the hydroxide of copper took place. This operation requires from one and a half to two hours, the preparation of larger quantities occupying a correspondingly larger amount of time. The solution was boiled some time to destroy the bicarbonate, cooled, and made up to the original volume.

Analyses show, as could be surmised beforehand, that this solution gives constant results; differences which could be ascribed to a varying carbonic acid content did not occur; we must naturally suppose that enough bicarbonate has been destroyed to prevent any crystallisation.

The following alternate methods of preparation were finally adopted as serving the purpose:—

A. To prepare 2 litres of the reagent, exactly 12.77 grammes of pure, finely powdered copper sulphate were dissolved in cold water, precipitated with soda lye, and the precipitate collected on a filter. A simple contrivance was used to facilitate washing. A flask holding about three-quarters of a litre was closed with a perforated stopper, through which passed a glass tube about 6 to 7 centimetres long and 5 millimetres wide. The flask was inverted in such a manner over the funnel that the end of the tube was just below the level of the liquid in the filter. This arrangement is automatic and reliable. The washed hydrate was carefully transferred to a porcelain dish, removing the last particles from the filter with the wash bottle, mixed well with a glass rod, and then washed into a 2500 c.c. flask together with 597.7 grammes potassium bicarbonate. Enough water was added to make the volume about 2000 c.c. This was heated on the water bath to 45° C., frequently shaking until the bicarbonate had dissolved, and then direct heat applied to dissolve the hydrate, not allowing the temperature to rise over 60° to 70° C. After complete solution the reagent is boiled for one to one and a half hours so as to decompose the bicarbonate, allowed to cool, transferred to a 2-litre flask, made up to the mark, and filtered. It is unnecessary to use a reflux condenser or to make up 2 litres beforehand, as the

escape of carbonic acid causes a concentration which must be corrected by the addition of water.

The solution in itself is stable, not even boiling causing decomposition. If, however, it is diluted with 5 times its volume of water a bluish green precipitate appears, which becomes black on boiling. This precipitate is therefore copper oxyhydrate, which is converted into black copper oxide on heating. This observation induced the author to examine the action of water on the usual Soldaini reagent. He found that when diluted with about 20 times its volume of water it also gave a precipitate of black oxide upon heating. This seems to indicate that the copper in Soldaini's reagent is not present as carbonate, but rather as hydrate.

B. The behaviour of basic carbonate of copper showed another way of preparing a constant reagent.

The basic carbonate prepared from 50 grammes copper sulphate is heated for five or six hours with 300 grammes potassium carbonate and about 700 c.c. of water, the heat being kept at 45° C. as before until the bicarbonate has dissolved and then at 60° C. The solution is filtered, and the excess of bicarbonate allowed to crystallise. The filtrate contained, in 100 c.c., 0.2882 grammes copper and a quantity of potassium hydrate, corresponding to 36.7 grammes potassium bicarbonate. To prepare 1 litre of Soldaini's reagent (0.1625 gramme Cu and 29.7 grammes HKCO_3 in 100 c.c.), 564 c.c. of above filtrate are required, which contain 207 grammes of the bicarbonate. It is therefore necessary to add 90 grammes HKCO_3 or an equivalent amount of K_2CO_3 , the use of the latter avoiding subsequent boiling for the purpose of destroying the excess of bicarbonate. A solution thus prepared could not be distinguished from another prepared by the first method. It depends altogether on the conditions whether one or the other should be preferred. If small quantities, for instance 2 litres, are to be prepared, the first is best applied; but if large quantities are required, as in manufacturing laboratories, where a steam bath is always at hand and the time is immaterial, then the last method is best followed.

2. *Ettore Soldaini's modification of Arturo Soldaini's solution.*—To avoid the decomposition of stronger solutions, E. Soldaini proposed the use of a deci-normal solution, made as follows:—3.464 grammes of crystallised copper sulphate ($\text{CuSO}_4 + 5 \text{H}_2\text{O}$) and 297 grammes of KHCO_3 are dissolved in a litre of water. For the estimation of the glucose with the $\frac{1}{10}$ normal solution 100 c.c. of this solution are heated to boiling, and the glucose solution added gradually until complete reduction takes place. The 100 c.c. of the $\frac{1}{10}$ normal solution correspond to 0.05 gramme of glucose.

The liability of Soldaini's reagent to decompose when much diluted explained the varying results obtained in the first investigations upon its reduction by invert sugar. Washing with hot water

at once induces this decomposition, to avoid which a washing with cold solution of potassium bicarbonate was substituted for the addition of water after boiling with the invert sugar until all the copper had been removed, following with water to remove all the alkali.

Ettore Soldaini prepares a deci-normal copper solution by dissolving 3.464 grammes CuSO_4 , 5 H_2O and 297 grammes KHCO_3 in 1000 c.c. of water. He deems it unnecessary to previously precipitate with Na_2CO_3 , and preferably adds the CuSO_4 solution, or even the powdered salt, to the solution of KHCO_3 . Sulphates exert no bad effect on the reagent, as might be inferred from the instructions of some to wash the copper carbonate precipitate. Experiments have shown this not to be so, and that 0.0005 gramme glucose dissolved in 10 c.c. can easily be detected when this is boiled for ten minutes with an equal volume of the reagent. The copper separates as intensely red suboxide. Under the above conditions less than the given amount of glucose cannot be detected. If, however, the test is made in the following manner, then 0.00025 gramme of glucose can easily be detected. The concentrated solution of the latter is mixed with just enough reagent to colour it slightly, and evaporated to dryness on the water bath. If glucose is present the porcelain dish will show a rose-coloured residue, which in the absence of glucose would be bluish green. Addition of water will show the colour more distinctly. To avoid mistakes, it is well to make a blank with the reagent similarly diluted.

The quantitative determination with this reagent ($\frac{N}{10}$) must be made as follows. Measure out 100 c.c. (corresponding to 10 c.c. of a solution containing 0.05 gramme pure glucose), heat until boiling, and add the unknown solution slowly until all the copper is reduced. When the approximate number of c.c. of the sugar solution required is thus determined the final estimation is made by boiling 100 c.c. of the reagent for five minutes in a 200 c.c. flask, adding the required volume of sugar solution from a burette, boiling fifteen minutes, and filtering after the liquid has been cooled rapidly. The filtrate should contain neither glucose nor copper.

This method gives such rapid and satisfactory results that Ettore Soldaini recommends the use of this $\frac{N}{10}$ copper solution.

3. *Scheller's modifications.*—Scheller states that Soldaini's reagent can be made of a definite composition by dissolving 15.8 grammes pure copper sulphate and 7.2 grammes potassium hydrate respectively in 100 c.c. of water, mixing and adding to 1700 c.c. of a solution of 594 grammes K_2CO_3 . It is then heated on the water bath until the precipitate dissolves, and after cooling made up to 2000 c.c. 1 c.c. of this reagent contains 2 milligrammes of copper. It is said to be constant and stable.

4. *Preuss's researches.*—Preuss emphasises the necessity, in determining the reducing power of invert sugar with Soldaini's reagent, of always using a reducing liquor of constant composition, *i.e.* having a certain percentage of copper and degree of alkalinity. Scheller's method recommends itself by its simplicity. It consists in adding 15.8 grammes CuSO_4 , 5 H_2O to a hot solution of 594 grammes K_2CO_3 , and making up to 2000 c.c., when the precipitate is dissolved. Three solutions made thus showed the same density 1.1789, a similar reducing power to invert sugars, and were very stable. As it is to be expected that the advantages which Soldaini's reagent possesses over Fehling's solution will cause its general adoption for the quantitative estimation of invert sugar, it will be of value to determine the exact reducing power of invert sugar with regard to this solution, especially as experiments so far made show it to be not exactly proportional to the quantity of invert sugar used. In the following experiments Soxhlet's 1 per cent. invert sugar solution was used, and the reduction made by heating 150 c.c. of the copper solution by the direct flame and adding the invert sugar from a pipette.

Upon boiling five minutes, 50 milligrammes invert sugar precipitates on an average 138 milligrammes copper; upon heating ten minutes, 156 milligrammes copper.

Period boiled.	Mg. Copper precipitated.
5	138
10	156
15	162
75	190
	75 mg. Invert Sugar.
15	231
	25 mg. Invert Sugar.
5	72
10	83
15	93
	40 mg. Invert Sugar.
5	112
10	127

This shows that a definite time of boiling must be observed in using Soldaini's reagent for quantitative purposes. As the separation of copper increases up to fifteen minutes' boiling, and, on the other hand, such long boiling renders analysis cumbersome and the solution of copper suboxide possible, ten minutes were taken as the basis for the determination of the curve of reduction. Under these conditions—

	Average.
I. 5 mg. invert sugar reduced	19 mg. Cu.
II. 10 mg. invert sugar reduced	34 mg. Cu.
III. 25 mg. invert sugar reduced	83 mg. Cu.
IV. 40 mg. invert sugar reduced	128 mg. Cu.
V. 50 mg. invert sugar reduced	157 mg. Cu.
VI. 60 mg. invert sugar reduced	184 mg. Cu.
VII. 70 mg. invert sugar reduced	214 mg. Cu.

Using these values, the following proportion is calculated for the curve of reduction by the method of the least squares—

$$y = 2.2869 + 3.299x - 0.004101x^2,$$

where x represents the milligrammes invert sugar used, and y the corresponding amount of copper. By solving the quadratic equation the invert sugar can easily be calculated from the copper found. The following table has been prepared to facilitate this :—

XI.

Mg. Invert Sugar.	Mg. Cu.	Mg. Invert Sugar.	Mg. Cu.	Mg. Invert Sugar.	Mg. Cu.	Mg. Invert Sugar.	Mg. Cu.
5	18.8	18	60.3	31	100.6	44	139.5
6	21.9	19	63.5	32	103.6	45	142.4
7	25.2	20	66.6	33	106.6	46	145.4
8	28.4	21	69.7	34	109.7	47	148.3
9	31.6	22	72.9	35	112.7	48	151.2
10	34.9	23	76.0	36	115.7	49	154.1
11	38.1	24	79.1	37	118.7	50	157.0
12	41.3	25	82.2	38	121.8	51	171.3
13	44.5	26	85.3	39	124.8	52	185.5
14	47.7	27	88.5	40	127.8	53	200.4
15	50.9	28	91.4	41	130.7	54	213.1
16	54.0	29	94.5	42	133.6	55	226.6
17	57.2	30	97.6	43	136.5	56	240.0

The values of the table correspond satisfactorily to the quantities of copper found experimentally, as the following shows :—

XII.

Mg. Invert Sugar.	Mg. Copper.	
	<i>Calculated.</i>	<i>By experiment.</i>
5	18.8	19
10	34.9	34
25	82.2	83
40	127.8	128
50	157.0	157
60	185.5	184
70	213.1	214

In carrying out the estimations of invert sugar by Soldaini's reagent, we should closely follow the method of manipulation laid down, using for reduction an amount of polarised filtrate correspond-

ing to 10 grammes of substance. The filtration must not be too slow, and too great a dilution must be avoided, so that no subsequent formation of copper oxyhydrate or cuprous oxide takes place.

Wilhelmy's researches.—The *time element in inversion* has been studied by Wilhelmy and others. The invertive power of very small quantities of mineral acids in concentrated sugar solutions is pointed out, especially with a mere trace of HCl, which, when melted with sucrose, secures a complete inversion thereof, and that too without noticeable discoloration. Illustrations are given of the action of HCl on concentrated sugar solutions.

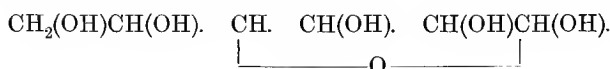
13.024 grammes of sucrose were heated for thirty minutes in a stoppered flask at 100°, and then showed a polarisation of 12°. With longer heating the left-handed polarisation diminished, and also the quantity of copper precipitated. It is concluded from the diminution of the left-handed polarising power of the invert sugar, caused by concentration of the solution and of the acid, and by high temperature and duration of the invertive process, that the invert sugar formed is not pure levulose and dextrose, but also contains a different sugar. If this diminution be due to a molecular condensation of the invert sugar formed, then it would take place also when pure invert sugar is heated with an acid. This experiment was tried with invert sugar and hydrochloric acid. Pure dextrose and levulose were used for these experiments. 3.425 grammes of each were taken and made up to 50 c.c. at 20°, polarising 15°.8. 0.1714 gramme of the mixed sugars gave 0.313 gramme copper. On heating this, as before, with dilute HCl, both polarisation and reduction were diminished. Heating with water alone gave no change, so that the condensation of the molecules of invert sugar is due to heating with acids.

It was necessary, in order to elucidate the problem further, to note the action of HCl on concentrated glucose and levulose solutions. These results showed that the dextrose was but little affected by the treatment, and hence that the changes noted had been due to polymerisation of the levulose.

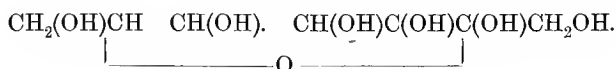
An examination of the products formed by the action of a small quantity of acid on concentrated levulose solutions shows that dextrin-like (levulin-like) bodies are formed (levulosin). An 80 per cent. solution of dextrose was heated at 105° with increasing quantities of acid. This treatment increased the polarisation of the substance, and diminished its reducing power, indicating a polymerisation and formation of dextrinoids. Whence it follows that the hydrolytic splitting up of the di- and poly-saccharides is not a simple process, but, together with the inverting action of the acid shown in the splitting up of the higher molecular weights, there is a synchronous reformation of the polymers. These phenomena give support to the opinion of Tollens, that the monoses are not pentaoyaldehydes or

pentaoxyketones, but pentaoxyoxides, that is, aldehyd or keto- γ -lactones, in which the structural formulæ for dextrose and levulose are as follows :—

Dextrose.



Levulose.



It is further shown that the usual methods of inversion use, without exception, excessive quantities of acid, and that the maximum levogyrous result and maximum reducing power are to be obtained only with very dilute acids or at low temperatures.

In order to convert pure sugar into colourless invert sugar, it is only necessary to heat the solution at 95° for half an hour with 0·01 per cent. HCl.

The above principles of inversion were also applied to starch, inulin, etc., on the supposition that the difficulty of securing a complete inversion of starch lay in the reformation of dextrin by the strong acid employed, rather than in the natural refractory nature of the material, and experiments are given supporting this hypothesis.

In the usual methods of inversion excessive quantities of acid are invariably used. The maximum levogyrous result and maximum reducing power are to be obtained only with very dilute acids or at low temperatures.

In order to convert pure sugar into colourless invert sugar, it is only necessary to heat the solution at 95° C. for a half-hour with 0·1 per cent. HCl. That the difficulty in securing a complete inversion of starch lies in the reformation of dextrin by the strong acid employed, rather than in the natural refractory nature of the material, has been demonstrated by experiment.

Maumené's theory.—According to Maumené, *invert sugar*, even when inverted by pure water, is never a mixture of equal weights of glucose and levulose, or even of chylariose. Glucose is a mixture of several hexeloses; one of these, left-handed hexelose, approaches very near in its composition to what is called levulose derived from inulin. It is very difficult to isolate it, even at a freezing temperature, and it is unstable in a higher temperature.

Another optically neuter, which has been called *mactose*, is obtained by a complete transformation of normal sugar by means of nitrate of silver. The third is a non-fermentable hexelose. It occurs in the inversion of normal sugar by pure water, or even by wate

which has been treated by a few ten-thousandths of acid. It is separated from the others by fermentation. The character of invert sugar is influenced—(1) by the relative quantities of sugar, water, and acid; (2) by the degree of heat; (3) by the time of the reaction; (4) temperature during the subsequent treatment with lime, saturation of the product by carbonic acid, and the duration of the evaporation *in vacuo*. Inversion is produced by a series of molecular movements modified by these numerous influences, and the greatest precautions are necessary to obtain the same result in like conditions. Glucose retains a certain quantity of a mixture of hexeloses, properly named *chylarose*, since it is extracted from the invert sugar by capillary attraction, porous brick or porcelain, or blotting paper. According to Maumené, Jungfleisch and Grimbert have confirmed the alteration of left-handed hexelose, not upon this hexelose properly so called, but on the inverted sugar, by a comparison with a product of inulin, a product which cannot be assimilated to invert sugar without many errors. The conclusions to which Jungfleisch and Grimbert came as the results of the experiments, to which Maumené takes exception, were—

1. Strong acids modify the rotary power of levulose, by increasing it more or less according to circumstances.

2. The levulose of sugar inverted by the processes in ordinary use is not strictly identical with pure crystallised levulose.

Jungfleisch and Grimbert's researches.—Some facts which have been developed in the experience of the authors lead them to doubt the principle of the method of Clerget. The process of Clerget may be expressed as follows:—

Let A be the deviation produced by 1 decimetre of the liquor in which it is desired to find the weights x and y of the sucrose and of the inverted sugar. Let A' represent the deviation of the same liquid inverted by strong acid without changing its concentration; then $(a)D = 67.31$ and $(a)D = -24.31$, this being the expression for the rotatory power of the sucrose and inverted sugar; then the two equations which will give the value of x and y are as follows—

$$\frac{67.31}{100}x - \frac{24.31}{100}y = A; \text{ and } \frac{24.31}{100}(x + y) = A'.$$

The results obtained in this way are generally inexact. Contrary to what is supposed in the first equation, inverted sugar exists in natural sugar products which have not been subjected to the action of strong acids; and its rotatory power, which at first is about $(a)D = 21.16$, increases greatly during inversion. Therefore the calculation indicates a plus error for the second, an error proportional to $\frac{y}{x}$, this increasing with the weight of inverted sugar pre-existing; moreover, the calculation exaggerates the error committed. It is

known that if such a sugar be treated with strong and dilute acids the product is always more strongly left-handed in the first than in the second case.

In the analysis of the crystallised sugar of commerce, which is always poor in inverted sugar, the difference is very little, and may be neglected; but such is not the case with sugars rich in inverted sugar. When the substances analysed contain glucose and levulose in unequal quantities, the other compounds are sometimes determined by the aid of an analogous calculation by estimating a third equation from the reduction of alkaline copper solution, but the same objection still applies. Acetic acid acts upon saccharose without modifying the levulose, but with some salts certain facts prevent its general use, namely—

1. Alkaline acetates, although they do not prevent the inversion of sucrose by strong acids employed in excess, interfere with the inversion by acetic acid even when employed in very great excess. In presence of 1 molecule of acetate of soda 1 molecule of sucrose is not completely converted, even at 100°, during half an hour by a weight of acetic acid equal to 80 molecules.

2. Alkaline citrates, formiates, lactates, and tartrates, the acetates of zinc and lead, act in a similar manner. The acetate of lime is much less active.

3. The salts of strong monobasic acids do not prevent inversion by acetic acid. The neutral salts of strong bibasic acetates diminish the inverting power when the base is monovalent, but not when the base is bivalent. The acid salts of strongly polybasic acids do not retard the inversion by acetic acid, but sometimes even affect it themselves.

GRAVIMETRIC ANALYSIS OF SUGAR

Soxhlet's gravimetric method.—It is important to always work under the same conditions: 60 c.c. of Fehling's solution, which does not require to be titrated, and which may be prepared there and then, is mixed with 60 c.c. of water. It is boiled, and all at once 25 c.c. of a solution, containing at the most 4 per cent. of sugar, is added, and after two minutes' boiling it is filtered through a special funnel of the same shape as the old calcium chloride tubes, 12 centimetres long and 13 millimetres in diameter, the bulb of which is half filled with asbestos fibres, not too soft, and laid crosswise as far as possible. The asbestos is washed with boiling water, dried, and the tube weighed exactly. It is then put in communication with an aspirating flask, and, with the aid of a funnel, the liquid with the precipitate is poured into the tube. The precipitate is first washed with boiling water, then with alcohol, then twice with absolute alcohol, and finally twice with ether. The ether is then driven off by means of a current of air. The lower orifice of the filtration tube is then connected with

a hydrogen-generating apparatus, the tube is inclined, and the bulb is heated by a small flame, care being taken to keep the point of it 5 centimetres below the bulb. In two or three minutes the reduction of the oxide of copper is complete. The tube is cooled in a current of hydrogen, a current of air is then passed through it, and the tube with its contents weighed.

The quantity of sugar may be calculated by aid of the following table :—

Copper in Milligrammes.	Glucose in Milligrammes.
10	6.1
20	11.0
30	16.0
50	25.9
100	50.9
200	102.6
300	156.5
400	212.9
463	249.9

XIII.—ESTIMATION OF INVERT SUGAR, CALCULATED FROM E. MEISSL'S
DATA BY E. WEIN.

Mg. Copper.	Mg. Invert Sugar.	Mg. Copper.	Mg. Invert Sugar.	Mg. Copper.	Mg. Invert Sugar.	Mg. Copper.	Mg. Invert Sugar.	Mg. Copper.	Mg. Invert Sugar.
90	46.9	113	59.1	136	71.3	159	83.8	182	96.2
91	47.4	114	59.6	137	71.9	160	84.3	183	96.8
92	47.9	115	60.1	138	72.4	161	84.8	184	97.3
93	48.4	116	60.7	139	72.9	162	85.4	185	97.8
94	48.9	117	61.2	140	73.5	163	85.9	186	98.4
95	49.5	118	61.7	141	74.0	164	86.5	187	99.0
96	50.0	119	62.3	142	74.5	165	87.0	188	99.5
97	50.5	120	62.8	143	75.1	166	87.6	189	100.1
98	51.1	121	63.3	144	75.6	167	88.1	190	100.6
99	51.6	122	63.9	145	76.1	168	88.6	191	101.2
100	52.1	123	64.4	146	76.7	169	89.2	192	101.7
101	52.7	124	64.9	147	77.2	170	89.7	193	102.3
102	53.2	125	65.5	148	77.8	171	90.3	194	102.9
103	53.7	126	66.0	149	78.3	172	90.8	195	103.4
104	54.3	127	66.5	150	78.9	173	91.4	196	104.0
105	54.8	128	67.1	151	79.4	174	91.9	197	104.6
106	55.3	129	67.6	152	80.0	175	92.4	198	105.1
107	55.9	130	68.1	153	80.5	176	93.0	199	105.7
108	56.4	131	68.7	154	81.0	177	93.5	200	106.3
109	56.9	132	69.2	155	81.6	178	94.1	201	106.8
110	57.5	133	69.7	156	82.1	179	94.6	202	107.4
111	58.0	134	70.3	157	82.7	180	95.2	203	107.9
112	58.5	135	70.8	158	83.2	181	95.7	204	108.5

[TABLE CONTINUED.]

XIV.—TABLE FOR USE IN THE GRAVIMETRIC ESTIMATION OF MILK SUGAR.

Mg. Cu.	Mg. Milk Sugar.	Mg. Cu.	Mg. Milk Sugar.	Mg. Cu.	Mg. Milk Sugar.	Mg. Cu.	Mg. Milk Sugar.	Mg. Cu.	Mg. Milk Sugar.
100	71·6	145	105·1	190	139·3	235	173·1	280	208·3
101	72·4	146	105·8	191	140·0	236	173·9	281	209·1
102	73·1	147	106·6	192	140·8	237	174·6	282	209·9
103	73·8	148	107·3	193	141·6	238	175·4	283	210·7
104	74·6	149	108·1	194	142·3	239	176·2	284	211·5
105	75·3	150	108·8	195	143·1	240	176·9	285	212·3
106	76·1	151	109·6	196	143·9	241	177·7	286	213·1
107	76·8	152	110·3	197	144·6	242	178·5	287	213·9
108	77·6	153	111·1	198	145·4	243	179·3	288	214·7
109	78·3	154	111·9	199	146·2	244	180·1	289	215·5
110	79·0	155	112·6	200	146·9	245	180·8	290	216·3
111	79·8	156	113·4	201	147·7	246	181·6	291	217·1
112	80·5	157	114·1	202	148·5	247	182·4	292	217·9
113	81·3	158	114·9	203	149·2	248	183·2	293	218·7
114	82·0	159	115·6	204	150·0	249	184·0	294	219·5
115	82·7	160	116·4	205	150·7	250	184·8	295	220·3
116	83·5	161	117·1	206	151·5	251	185·5	296	221·1
117	84·2	162	117·9	207	152·2	252	186·3	297	221·9
118	85·0	163	118·6	208	153·0	253	187·1	298	222·7
119	85·7	164	119·4	209	153·7	254	187·9	299	223·5
120	86·4	165	120·2	210	154·5	255	188·7	300	224·4
121	87·2	166	120·9	211	155·2	256	189·4	301	225·2
122	87·9	167	121·7	212	156·0	257	190·2	302	225·9
123	88·7	168	122·4	213	156·7	258	191·0	303	226·7
124	89·4	169	123·2	214	157·5	259	191·8	304	227·5
125	90·1	170	123·9	215	158·2	260	192·5	305	228·3
126	90·9	171	124·7	216	159·0	261	193·3	306	229·1
127	91·6	172	125·5	217	159·7	262	194·1	307	229·8
128	92·4	173	126·2	218	160·4	263	194·9	308	230·6
129	93·1	174	127·0	219	161·2	264	195·7	309	231·4
130	93·8	175	127·8	220	161·9	265	196·4	310	232·2
131	94·6	176	128·5	221	162·7	266	197·2	311	232·9
132	95·3	177	129·3	222	163·4	267	198·0	312	233·7
133	96·1	178	130·1	223	164·2	268	198·8	313	234·5
134	96·9	179	130·8	224	164·9	269	199·5	314	235·3
135	97·6	180	131·6	225	165·7	270	200·3	315	236·1
136	98·3	181	132·4	226	166·4	271	201·1	316	236·8
137	99·1	182	133·1	227	167·2	272	201·9	317	237·6
138	99·8	183	133·9	228	167·9	273	202·7	318	238·4
139	100·5	184	134·7	229	168·6	274	203·5	319	239·2
140	101·3	185	135·4	230	169·4	275	204·3	320	240·0
141	102·0	186	136·2	231	170·1	276	205·1	321	240·7
142	102·8	187	137·0	232	170·9	277	205·9	322	241·5
143	103·5	188	137·7	233	171·6	278	206·7	323	242·3
144	104·3	189	138·5	234	172·4	279	207·5	324	243·1

[TABLE CONTINUED.]

XIV.—GRAVIMETRIC ESTIMATION OF MILK SUGAR—*continued.*

Mg. Cu.	Mg. Milk Sugar.	Mg. Cu.	Mg. Milk Sugar.	Mg. Cu.	Mg. Milk Sugar.	Mg. Cu.	Mg. Milk Sugar.	Mg. Cu.	Mg. Milk Sugar.
325	243·9	341	256·5	356	268·8	371	281·4	386	294·2
326	244·6	342	257·4	357	269·6	372	282·2	387	295·1
327	245·4	343	258·2	358	270·4	373	283·1	388	296·0
328	246·2	344	259·0	359	271·2	374	283·9	389	296·8
329	247·0	345	259·8	360	272·1	375	284·8	390	297·7
330	247·7	346	260·6	361	272·9	376	285·7	391	298·5
331	248·5	347	261·4	362	273·7	377	286·5	392	299·4
332	249·2	348	262·3	363	274·5	378	287·4	393	300·3
333	250·0	349	263·1	364	275·3	379	288·2	394	301·1
334	250·8	350	263·9	365	276·2	380	289·1	395	302·0
335	251·6	351	264·7	366	277·1	381	289·9	396	302·8
336	252·5	352	265·5	367	277·9	382	290·3	397	303·7
337	253·3	353	266·3	368	278·8	383	291·7	398	304·6
338	254·1	354	267·2	369	279·6	384	292·5	399	305·4
339	254·9	355	268·0	370	280·5	385	293·4	400	306·3
340	255·7								

XV.—GRAVIMETRIC ESTIMATION OF SMALL QUANTITIES OF GLUCOSE (HERZFELD).

Copper Mg.	Invert Sugar Per Cent.	Copper Mg.	Invert Sugar Per Cent.	Copper Mg.	Invert Sugar Per Cent.
50	0·05	140	0·51	230	1·02
55	0·07	145	0·53	235	1·05
60	0·09	150	0·56	240	1·07
65	0·11	155	0·59	245	1·10
70	0·14	160	0·62	250	1·13
75	0·16	165	0·65	255	1·16
80	0·19	170	0·68	260	1·19
85	0·21	175	0·71	265	1·21
90	0·24	180	0·74	270	1·24
95	0·27	185	0·76	275	1·27
100	0·30	190	0·79	280	1·30
105	0·32	195	0·82	285	1·33
110	0·35	200	0·85	290	1·36
115	0·38	205	0·88	295	1·38
120	0·40	210	0·90	300	1·41
125	0·43	215	0·93	305	1·44
130	0·45	220	0·96	310	1·47
135	0·48	225	0·99	315	1·50

XVIA.—GRAVIMETRIC DETERMINATION OF GLUCOSE IN SUGARS CONTAINING MORE THAN 1½ PER CENT. (MESSL).

Ratio of the Two Sugars.	Invert Sugar in Milligrammes.								
	245	225	200	175	150	125	100	75	50
Crystal to Invert Sugar.	F.	F.	F.	F.	F.	F.	F.	F.	F.
90 : 10 . . .	56·2	55·1	54·1	53·6	53·1	52·6	52·1	51·6	51·2
91 : 9 . . .	56·2	55·1	54·1	53·6	52·6	52·1	51·6	51·2	50·7
92 : 8 . . .	56·2	54·6	53·6	53·1	52·1	51·6	51·2	50·7	50·3
93 : 7 . . .	55·7	54·1	53·6	53·1	52·1	51·2	50·7	50·3	49·8
94 : 6 . . .	55·7	54·1	53·1	52·6	51·6	50·7	50·3	49·8	48·9
95 : 5 . . .	55·7	53·6	52·6	52·1	51·2	50·3	49·4	48·9	48·5
96 : 4	52·1	51·2	50·7	49·8	48·9	47·7	46·9
97 : 3	50·7	50·1	49·8	48·9	47·6	46·2	45·1
98 : 2	49·9	48·9	47·3	47·3	45·8	43·3	40·0
99 : 1	47·7	47·3	45·1	45·1	43·3	41·2	38·1

Fehling's solution.—CuSO₄, 5 Aq 34·64 grammes; Rochelle salt, 173 grammes; caustic soda solution, 480 c.c.; distilled water to 1 litre.

XVII.—COMPARISON OF VIOLETTE'S AND FEHLING'S SOLUTION.

Sugar.	10 c.c. Fehling's Solution correspond to	10 c.c. Violette's Solution correspond to
Cane sugar . . .	0·0475	0·050
Dextrose . . .	0·048	0·051
Invert sugar . . .	0·050	0·053
Levulose . . .	0·052	0·055
Lactose . . .	0·067	0·072
Maltose . . .	0·075	0·079

New test for cane sugar.—2 c.c. of the sugar solution, 5 drops of a 20 per cent. alcoholic solution of *a*-naphthol, and 10 c.c. pure conc. H₂SO₄ are mixed in a test-tube. The liquid is coloured violet or bright rose, according to the percentage of sugar. The reaction is very decided with 1 part of sugar in 100,000 of water, and even smaller quantities may be detected. Pellet has introduced a quantitative process based on this reaction.

CHAPTER II

BEEET SUGAR

PURCHASE AND ANALYSIS OF BEETS

PURCHASE of beets by contract.—*The contract specifications.*—Sugar beets are sold by contract, *i.e.* the farmer agrees to grow sugar beets under predetermined conditions, at a price fixed beforehand. At the time of laying down the crop the manufacturer endeavours to make himself sure of getting prompt delivery of a certain quantity of roots, so as to enable him to negotiate on a certain quantity of sugar, to be delivered after manufacture. The farmer, on his part, seeks an outlet for his crop. They enter into a mutual agreement, by which the farmer, on his part, engages to deliver, beginning with a stated period, sugar beets from such and such a field, of a determined composition at the price fixed beforehand. The farmer is generally free to till the ground as he likes, and to use whatever manure suits him best. The manufacturer supplies the seeds, so as to ensure the authenticity and value of the varieties cultivated.

The farmer's risk in waiting to secure the current market price.—Some farmers do not enter into an agreement as to the delivery of beetroots to the factory. They prefer to wait until the sales in the month of September, so as to discuss the price of their beets by the prices ruling in the sugar market. But in these cases they may be at the mercy of the manufacturer if they do not wish to risk their crop. It is therefore prudent for them to sign an agreement.

Delivery.—The roots are often carted to the factory, but, unless the fields be quite near, that is a source of great expense, which certain factories endeavour to reduce by establishing depôts at their own cost, provided with steelyards, near to the farms and on the most frequented highways. In some cases the beets are brought by rail or boat. The methods of transporting the beets from the depôts to the factory will be discussed later on.

Sampling.—The deliveries are sampled in the cart, in the railway waggon, and, in certain cases, in the field, or in the pit or silo.

Sampling in the cart or railway waggon.—When the sugar beets are sold in the railway waggon or cart, a determination of the density or the richness in sugar is made on each waggon, or every two or three

carts, taking care to choose the roots from the whole of the bulk, so as to obtain a fair average sample.

In the field.—The samples are sometimes taken in the field. A certain number of roots are pulled, which are arranged in lines or rows, in the order of their size; then the tenth, the twentieth, etc., is taken, so as to get twenty-five to thirty roots per hectare (say ten to twelve per acre). The sampling may be done in a similar way, before pulling the roots, by starting in a line, chosen at random, and proceeding in a straight line.

Sampling the deliveries at the factory.—The manufacturer always reserves the right of controlling the density, as per sale contract, by taking samples at the time of delivery of the roots to the factory.

Tare (allowance to be deducted for mud).—A certain amount of soil always adheres to the beets at the time they are pulled, more especially after rain, when they sometimes bring with them a considerable weight of soil. The weight of this mud must be deducted from that shown on the steelyard. To ascertain exactly the weight of roots available for manufacture, a certain weighed quantity is taken at the time of delivery, say a basketful of beets from all over a cart. These beets are weighed and then washed in a drum with rotating blades, then scraped, brushed, and reweighed, and the difference gives the mud which the beet has brought along with it, and is called the tare.

Heading and tailing the beets at the factory.—Before the second weighing, however, care is taken to strip the beet of any remaining leaves, and, unless the operation has been previously satisfactorily performed on the field, the beets are “headed” again, *i.e.* the top of the beet is cut off flat, perpendicular to the axis at the base of the first leaf. This operation should of course be done on the field, when the beets are pulled up. This is an important matter, as the top conical part of the beet, which bears the leaves, contains but little sugar, and is, moreover, highly charged with injurious salts. Therefore, if the heading has not been satisfactorily done on the field, the operation is completed at the factory. The beets which have been cleaned, and weighed to get at the tare, are used for the analysis, when the beets are bought according to their sugar content. By a recent legislative enactment, all the steelyards used in the weighing of beets in France must be furnished with a registering apparatus, working in such a way as to successively print, on the same ticket and on lines sufficiently far apart, the weight of the cart when full and when empty.

These operations, relative to the weighing, the waste, and the density, ought, in France, to take place under the control of the exciseman attached to the factory. The manufacturers are bound to facilitate this control and to provide the necessary instruments. But by a tacit agreement between the farmers and the manufacturers there are but few factories which conform to these arrangements for the reception of

beetroots by the Excise officials. The Excise arrangements for weighing the beets as they pass in for treatment are detailed on p. 73 *et seq.*

Sale of beets by analysis.—It is quite natural that the basis of the sale of beets should depend on their richness in sugar. In order to determine this, three methods may be adopted—

1. The determination of the density of the juice.
2. The saccharimetric value of the juice.
3. The direct analysis of the pulp.

Estimation of the percentage of sugar in beets from the density of the expressed juice.—Beets are purchased by the density of the juice in France since the legislative enactment of 1884 came into force, although it is a well-known fact that there is no well-determined ratio between (1) the density of the juice, and (2) the actual percentage of sugar in the beet. This ratio may, in fact, vary from 1.75 to 1.97 per unit of density, the sugar being of course calculated on the weight of the root. When it is a case of determining the sugar content of a beet, so as to decide whether it should be selected as a seed producer, polarimetric methods (Fig. 7) must be adopted.

XVII.—TABLE OF CORRECTIONS TO BE MADE ON THE APPARENT DENSITY OF BEET JUICE ACCORDING TO THE TEMPERATURE.

Temperature °C.	To be deducted.	Temperature °C.	To be added.
0	0.20	16	0.02
1	0.19	17	0.05
2	0.18	18	0.07
3	0.17	19	0.10
4	0.16	20	0.12
5	0.15	21	0.15
6	0.14	22	0.17
7	0.13	23	0.20
8	0.12	24	0.22
9	0.11	25	0.25
10	0.10	26	0.28
11	0.09	27	0.31
12	0.07	28	0.34
13	0.05	29	0.37
14	0.02	30	0.40
15	0.00	31	0.43
		32	0.46
		33	0.49
		34	0.52
		35	0.55
		36	0.60
		37	0.64
		38	0.67
		39	0.70
		40	0.74

Influence of temperature on density.—The density is taken by means of a hydrometer, which marks 0° in distilled water at 15° C. If the operation be not conducted at this temperature a correction is necessary, because this liquid expands and becomes lighter as the temperature rises, and *vice versa*.

Tables have been constructed giving the corrections to be made, but they cannot be quite exact, as the expansion of the juice varies with its composition. It has been suggested that it would be sufficient to add or deduct 0.1 from the gravity found, according as the temperature is 4° above or below 15° C.

The use of the above table, however, has been made obligatory in France in the case of beet deliveries by legislative enactment, promulgated on the 23rd July 1897.

Explanation of table.—Suppose the hydrometer shows a density of 1.075, which is called 7.5 Excise degrees. Let the temperature by the Centigrade thermometer be 19° : 19 is found in the temperature column, and in the other column it will be seen that it is necessary to add 0.1 Excise degree to the result found, which gives for the density of the juice—

$7.5 + 0.1 = 7.6$ Excise degrees, or an actual density of 1.076.

Suppose, on the other hand, that the density found is 1.085, which is announced as 8.5 at the temperature of 9° C., the corrected density will be—

$8.5 - 0.11 = 8.39$ Excise degrees, or an actual density of 1.0839.

According to contracts, if it be a case of purchase the density will be taken as 8.3 or 8.4 instead of 8.39. The determination of the density of the juice, although at first sight an extremely simple operation, requires certain precautions to be observed, so that the readings of the hydrometer may be exact.

Preparing the samples of beets.—The first thing to be done is to well clean and head the beets which form the lot to be analysed, and which should not be less than five in number. If the lot to be analysed is not a big one, the whole of the roots are pulped. Otherwise only half or quarter of the roots, cut exactly in the direction of their length, are pulped, placed in a stout calico bag, and strongly pressed in a screw press with a spout, through which the juice flows into a collecting vessel. The delivery is then paid for according to their density, an average rate as a basis of price, for example, being 25 francs (say £1) per metric ton of beets of 6° (that is, sp. gr. 1.06), and reducing or increasing this price for each tenth of a degree greater or less.

The first and final flow differ in density.—It would appear to result, from experiments recently made by Saillart at the French National School of Agricultural Industry, that the first flow of juice is always of greater density and is richer in sugar than the final flow. The variation may be as much as $\frac{2}{10}$. The juice is poured into a

tall cylindrical jar, wide enough for a hydrometer to float in it easily. It is necessary to fill the jar to the brim, so that the liquid flows over, so as to get rid by blowing of the froth on the surface, and to wait ten to twelve minutes before inserting the hydrometer, so that no more froth may be formed. It is due, in fact, to air-bells in suspension in the liquid, which may reduce the density by several tenths of a degree.

The correct method of reading the hydrometer meniscus.—When the hydrometer is immersed in the liquid the latter rises against the sides of the jar, forming what is called the *meniscus*. The reading should not be taken from the top of the attenuated layer around the stem, but on the prolongation of the horizontal surface of the liquid, which it is always easy to discern, with sufficient approximation, in graduated hydrometers.

A prevalent fallacy and its effects.—Certain farmers have got the wrong idea that the percentage richness in sugar of the beet may be obtained by multiplying the density by 2. This is quite wrong.

However, starting from this idea that the percentage of sugar was double the degree, a custom has been established in France whereby the percentage of sugar divided by 2 was taken as the density. The error arose from a loose habit, formerly contracted by chemists, of inserting in their reports of the indirect analyses of beet the percentage of sugar in c.c. of juice—figure of no great value—and of dividing this result by the density. Coefficients were thus obtained varying from 2 to 2.2, which amounts to saying that in 100 c.c. of juice extracted from the beets in question there was a quantity of sugar equal to 2 to 2.2 times the density.

The percentage of sugar in juice of given density not always uniform.—Moreover, the same density is far from corresponding to the same percentage of sugar, and the error in the system may amount to as much as two units. The weight of the sugar per cent. in the sugar beet is, on an average, inferior or superior to the degree multiplied by 2, as that degree is itself lower or higher than 7.5.

Density by itself an insufficient guide.—The density by itself alone, therefore, is not sufficient to give an exact idea of the composition of the sugar beet. However good it may be for giving the farmer some information as to the quality of his beets, it furnishes but an uncertain basis for contracts.

Simplicity its chief recommendation.—An argument, however, in favour of this method is its simplicity. It requires no laboratory apparatus; moreover, it has the further recommendation of both contracting parties being able to follow and understand its operations, which can be executed and discussed under the eyes of those interested.

Saccharimetrical valuation.—Sometimes the juice is tested optically by the saccharimeter, and it is on the polarisation results

that the farmer is paid. But this is the exception and not the rule, as the farmer will have nothing to do with the results of an instrument the use of which he in no way understands.

XVIII.—ESTIMATION OF THE AMOUNT OF SUGAR IN BEETROOTS
(EDOUARD DELVILLE).

Density.	Factor.	Density.	Factor.	Density.	Factor.
1·04	0·164	1·057	0·162	1·074	0·159
1·041	0·164	1·058	0·162	1·075	0·159
1·042	0·164	1·059	0·161	1·076	0·158
1·043	0·164	1·06	0·161	1·077	0·158
1·044	0·163	1·061	0·161	1·078	0·158
1·045	0·163	1·062	0·161	1·079	0·158
1·046	0·163	1·063	0·160	1·08	0·158
1·047	0·163	1·064	0·160	1·081	0·158
1·048	0·163	1·065	0·160	1·082	0·158
1·049	0·163	1·066	0·160	1·083	0·157
1·05	0·163	1·067	0·160	1·084	0·157
1·051	0·162	1·068	0·159	1·085	0·157
1·052	0·162	1·069	0·159	1·086	0·157
1·053	0·162	1·07	0·159	1·087	0·157
1·054	0·162	1·071	0·159	1·088	0·157
1·055	0·162	1·072	0·159	1·089	0·156
1·056	0·162	1·073	0·159		

This table is intended for use with saccharimeters of the Duboseq-Soleil type, with a normal weight of 16·35 grammes, and supposing the beetroot to contain 0·95 of its weight of juice, the figure on which transactions are generally based.

In order to calculate the percentage of sugar by means of this table, two determinations are required—(1) The density of the juice at 15° C.; (2) the polarisation of the juice after adding $\frac{1}{10}$ of basic acetate of lead. These having been made, the density is sought in the table, and the number of degrees on the scale of the saccharimeter is multiplied by the factor opposite the density. The product gives the percentage of sugar in the beet.

Example.—The juice marks 1°·05 density. The saccharimeter shows 60°. The percentage of sugar in the beet is $0·163 \times 60 = 9·78$ per cent.

XIX.—DENSITY OF SOLUTIONS OF CANE SUGAR IN WATER AT 15° C.
 (SCHEIBLER).

Per Cent.	Density.	Per Cent.	Density.	Per Cent.	Density.	Per Cent.	Density.
0	1·00000	19	1·07907	38	1·16960	57	1·27297
1	1·00390	20	1·08354	39	1·17470	58	1·27879
2	1·00783	21	1·08804	40	1·17985	59	1·28465
3	1·01178	22	1·09257	41	1·18503	60	1·29056
4	1·01576	23	1·09713	42	1·19024	61	1·29650
5	1·01978	24	1·10173	43	1·19550	62	1·30248
6	1·02382	25	1·10635	44	1·20079	63	1·30850
7	1·02789	26	1·11101	45	1·20611	64	1·31457
8	1·03199	27	1·11571	46	1·21147	65	1·32067
9	1·03611	28	1·12044	47	1·21687	66	1·32682
10	1·04027	29	1·12520	48	1·22231	67	1·33301
11	1·04446	30	1·12999	49	1·22779	68	1·33923
12	1·04868	31	1·13482	50	1·23330	69	1·34550
13	1·05293	32	1·13969	51	1·23885	70	1·35182
14	1·05721	33	1·14458	52	1·24444	71	1·35817
15	1·06152	34	1·14952	53	1·25007	72	1·36457
16	1·06586	35	1·15448	54	1·25574	73	1·37101
17	1·07023	36	1·15949	55	1·26144	74	1·37749
18	1·07464	37	1·16452	56	1·26718	75	1·38401

The analysis of the expressed juice or the indirect analysis of beet.
 —The percentage of sugar in the juice may be determined very accurately by the saccharimeter: 100 c.c. of the juice used for the density determination are run into a flask, graduated in two places—one at 100 c.c. and the other at 110 c.c. The solution is made up to 110 c.c. by the addition of 10 c.c. of basic acetate of lead of 28° or 30° Baumé. The flask is agitated and left to stand until some colourless points are perceived in the greenish mass. It is then filtered, and the filtrate examined in a 200-millimetre saccharimetric tube. If the solution is cloudy, it is clarified with a drop or two of acetic acid.

The sugar per 100 c.c. of juice is obtained by increasing the result by $\frac{1}{10}$, to make allowance for the basic acetate of lead (see Table XX.).

The richness of the juice does not, however, represent that of the beet, and it is the latter that the manufacturer wants to know. To ascertain which, it is taken for granted that the amount of juice contained in the root is somewhere about 95 per cent. of its weight, and that the juice extracted from the pulp by pressure is similar enough to that left behind. But two causes of error may influence the result. First of all, the proportion of juice is not always exactly 95 per cent., and, again, it has been demonstrated that the juice obtained by pressure is richer in the beginning of the flow than at the end. This was pointed out in 1896 by Pagnoul, who at the same time showed that the juice, whilst it became less rich, became also more pure, which explains why the density decreases with greater speed than the richness. There is therefore no homogeneity between the extracted juice and the total juice contained in the root. In order to deduce the percentage of sugar in the beet from that in the juice, it is necessary to use a coefficient which represents the proportion of juice in 100 of pulp.

To find this coefficient, it is first of all necessary to determine the percentage of sugar in the juice, then, by a direct analysis, that in the pulp, and finally, by means of the formula which we have indicated, the number by which it is necessary to multiply the first to get the second. In 82 comparative analyses on pulp and juice Pagnoul found this coefficient in 78 cases included between 91 and 95, and grouped around 93. In no case did he find a lower figure than 90. He considers that it would be desirable in indirect analyses to adopt the coefficient 93, not as representing the quantity of juice per cent. contained in the beet, but to pass from the richness of the juice to that of the beet.

Influence of the precipitate formed by acetate of lead upon polarisation.—Let S equal the weight of 100 c.c. of juice; B, the weight of 10 c.c. acetate of lead solution; N, the weight of the precipitate formed; F, the weight of the filtered liquid; then $S + B = N + F$. If for F we substitute the product of the volume V by the density d , we obtain $S + B = N + Vd$. If the volume of the precipitate be represented by v , we have the equation $V + v = 110$, whence $V = 110 - v$. From these equations the following one is derived—

$$v = \frac{110d + N - (B + S)}{d}$$

From this formula is easily deduced the volume of the lead precipitate.

Example.—Weight of 100 c.c. of juice plus 10 c.c. of lead acetate = 118.509 grammes = $S + B$. Density of filtered liquid = $1.061 = d$. The weight of the dried precipitate was found to be $3.005 = N$, then by the formula above $N = 1.136$. It is thus seen that the lead precipitate occupies a little more than 1 per cent. of the total volume.

Purity.—From the density and the percentage of sugar in the juice the *purity* may be deduced, that is to say, the relation between the *weight p of the sugar contained in 100 c.c. of the juice*, and the *weight P which 100 c.c. of a solution of pure sugar should contain in order to have the same density*. The purity will therefore be expressed by the equation $\frac{p}{P}$, and the difference, $P - p$, will be what is called *non-saccharine*. The non-saccharine is therefore got by deducting the polarisation sugar from the figures representing the dissolved matter, both sugar and non-saccharine being calculated on the same basis of 100 c.c. or 100 grammes. The quotient of purity represents the percentage of sugar in the dissolved matter. It is ascertained by the formula—

$$Q = \frac{100 S.}{\text{Dissolved matter}}$$

To get the weight P, the tables which give for each degree of density the weight of sugar necessary to make 100 c.c. of the solution must be consulted. According to Pagnoul, the figures contained in these tables may be approximately obtained by multiplying the French Excise degree by the coefficient 2.6. (See pp. 3, 4.)

THE DIRECT ANALYSIS OF THE SUGAR BEET

The difficulties in obtaining invariable results by operating on the pressed juice.—Reference has already been made to the fact that when beet pulp is pressed under great pressure the nature of the juice which exudes at the beginning is not the same as that which exudes at the finish, the final juice not being so dense as the initial. It therefore follows that if the pulp be pressed (*a*) by hand, (*b*) under an ordinary hand press, (*c*) under a powerful hydraulic press, each of the three methods will yield a different liquid.

However, if in a sugar factory it be customary to use a hand press, which is always turned by the same individual, who as a rule always exerts the same pressure, then the results are comparative, so far as that factory is concerned, although they are not so with those of a neighbouring factory using a hydraulic press.

Preparation of pulp for analysis.—In the factory pulping of beets for analyses Pellet's pulper shown in Fig. 4 is used. In its present general form it was in use prior to his aqueous digestion process about to be described. It was primarily intended for the pulping of beets, the juice of which was to be extracted by pressure. It was intended to be used in getting a slice throughout the whole of the length of any given beet from the centre to the circumference, and radiating under a given fixed angle representing about $\frac{1}{8}$ of the beet to be analysed. The cutting is therefore done at an angle of about 30° throughout the whole length of the beet.

He accordingly made a disc, thick in the centre and diminishing to zero at the circumference. This disc was provided with teeth; and it is evident that, in turning it, if a beet were passed in from the head to the tail, and going as far as the centre, a slice will have been cut over the whole of its half surface which will be of the same shape and the same relation as that made by any other beet presented to the instrument in the same way.

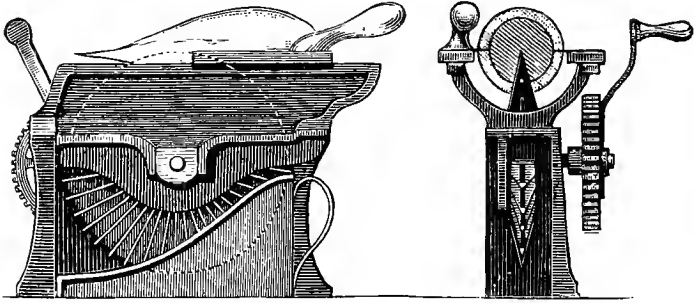


FIG. 4.—Pellet's beet pulper for preparing samples of beets for analysis.

It is Pellet's disc, dressed like a saw for wood, which is used in factories when there is a large quantity of beets to pulp for analysis.

In the analysis of beets selected for seed producers use is made of a pulp drill dressed in the same way, only, as the pulp thus formed has to be collected in the hole made by the drill in the beet, the

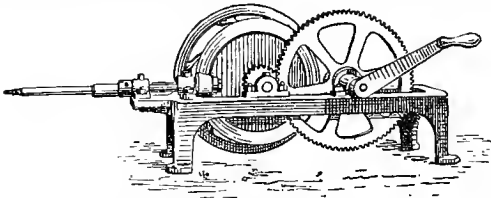


FIG. 5.—Mechanical drill for sampling beets.

revolving rod of the drill, and the drill, itself is hollowed in such a way that the rasped pulp traverses the drill and is lodged in the hollow rod. When the hole is pierced the rod is withdrawn, the pulp in the interior extracted, and the sample for analysis is obtained.

Hanriot's apparatus.—Fig. 6 consists of a truncated steel cone Keil-dressed, turning vertically, and against which the small pieces of beet taken out by the drill from the mother-root are pressed. The

truncated cone is adjusted to a frame carrying the driving gear A, the pusher, and a caoutchouc pear P for the wash-water, the whole within a small compass so as to facilitate operations. The cylindrical slice obtained by drill shown in Fig. 5 is weighed, and an exact length giving the desired weight is measured and introduced into the pulper, and its pulp washed into the 50 c.c. flask. The apparatus is then washed with water, the washings falling into the flask in such a

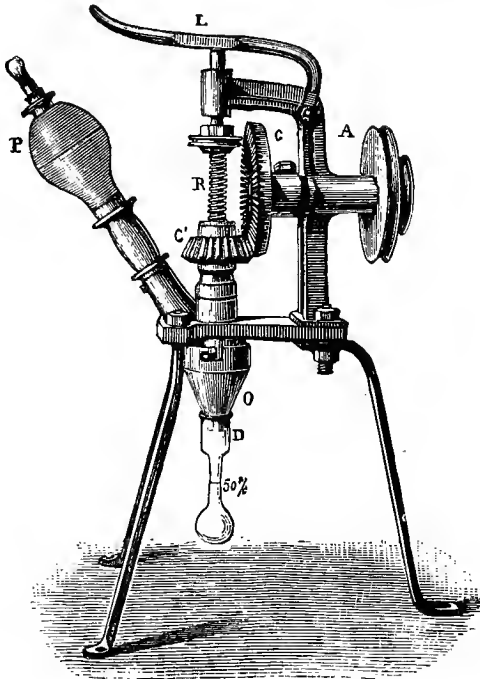


FIG. 6.—Hanriot's apparatus for pulping beet samples.

way that these preliminary operations are done in the twinkling of an eye.

Pellet's adaptation.—To avoid weighing the cylinder, *Pellet* proposes to use a knife with two parallel blades capable of being regulated. He bases this process on the fact that the difference in weight between two small cylinders of beetroot of the same diameter and of the same determined length is unappreciable.

Hanriot's apparatus (Fig. 6) can pulp 2200 and even 3000 cylinders a day. A knife can cut 15,000 to 18,000 cylinders per ten hours' day.

Care should be taken in using Hanriot's apparatus to collect the juice in a 200 c.c. flask instead of 100, which thus diminishes the strength of the liquid by one-half. Frothing is thus prevented, but then the result must be doubled. But it is preferred to use a saccharimetric tube of double the length, so as to be more exact and avoid calculation.

The direct analysis of the pulp of the sugar beet by Pellet's aqueous digestion method is to be preferred to the analysis of the expressed juice.

Pellet's hot aqueous digestion process.—*Principle on which it is based.*—This process is based on the fact that all the sugar in the more or less finely divided pulp is entirely soluble in hot water of 70° to 80° C., the digestion being continued for a quarter of an hour to one hour. The extract is cooled and made up to a given volume, taking into account not only the volume occupied by the insoluble marc, but also that occupied by the precipitate produced by the basic acetate of lead added before digestion, so as to precipitate a large proportion of the extraneous matter, which possesses a polarising power of 3·75, sugar being taken as unity.

The hot aqueous method is a very exact method, but not quite simple enough for certain purposes. When a cream pulp is to be tested, the cold digestion process is sufficient to dissolve all the sugar.

Pellet's hot diffusion process.—A certain weight of the pulp produced by a Pellet and Lomont's pulper (Fig. 4), and made homogeneous by stirring, is weighed out, say 16·29 grammes or 32·58 grammes for Laurent's saccharimeter. The pulp is then transferred to a 100 or 200 c.c. flask, with 5 to 6 c.c. of a solution of basic acetate of lead of 28° to 30° Baumé. It is made up almost to the mark with water, and kept in a water bath at a temperature of 80° C. for 30 to 45 minutes. The cooled liquid, to which a drop of ether has been added to disperse the froth, is made up with water to 100·9 or 200·7 c.c., so as to take the insoluble into account. Sidersky allows 200·8 c.c. for 16·29 grammes, and 201·35 c.c. for 26·048 grammes, and if the flask be not graduated to these marks he takes 16·19 instead of 16·29 grammes, and 25·87 instead of 26·048 grammes. The flask is shaken, then its contents are passed through a filter; after throwing away the first drops, which are generally turbid, a clear liquid is obtained, with which the 20 c.c. polarimeter tube is filled. The reading of the saccharimeter gives directly the percentage of sugar.

Pellet's instantaneous cold diffusion process.—This method is now very generally adopted, both on account of its simplicity and the correctness of the results obtained. It is generally recognised as the most practical of all methods, whilst it gives results equal, if not superior, in accuracy to the most intricate of previous methods. The

principles of the process are as follows. All the sugar present in sugar-beet pulp of a certain degree of fineness, such as is obtained by passing it over a somewhat blunt saw, is instantaneously diffused through water in sufficient quantity, and immediately dissolved therein in presence of basic acetate of lead. The cells in this case appear to be completely torn asunder to such an extent that their saccharine juice dissolves immediately in water. There is no diffusion, properly so-called—simply dilution. To apply this process to the analyses of individual sugar beet, a laboratory fitted up with the following appliances is required :—

1. One or more pigeon-hole cases with numbered receptacles in which the beets to be analysed are kept.

2. A drill-pumping machine, driven by hand or motor power at a speed of 2000 revolutions a minute, to produce a very fine pulp called pulp cream.

3. One or more balances or scales sensible to 1 centigramme.

4. Tables, graduated 50 or 100 c.c. flasks, basins, funnels, nickel cups, test glasscs, numbered copper forceps, filter paper.

5. A saccharimeter, working white or yellow light according to the method of illumination adopted.

Process.—A special workman presents each beet to the drill about the upper third of its height, inclining it slightly but not pressing it. The beet, drilled from side to side, is passed to an assistant, who places it in a case, with a label bearing a consecutive number; the drill having been withdrawn, the pulp is cautiously extracted and placed in a numbered saucer, which is handed to the weigher. The latter mixes it intimately, and weighs out from it a certain quantity, which is transferred, by means of water and through a wide-necked funnel, into a graduated flask. A solution of basic acetate of lead of 30° B. is added to refine the juice. The volume is then made up with water to the graduated mark. If need be, the froth is dispersed by a few drops of ether. The flask is shaken, then its contents are filtered, and the filtrate, clarified if need be with a few drops of acetic acid, is polarised in a 400-millimetre tube, and the direct percentage of sugar in the pulp is soon ascertained. When a large number of samples have to be tested, the continuous flow tube (Fig. 7) may be used.

The figures showing the percentage of sugar in each root are noted at the conclusion of each test in a special register, and opposite each analysis the weight of each beet recognised as good is placed. This weight is taken on a special balance.

To avoid the fatiguing reading of the degrees, an electrical arrangement may be fitted to the saccharimeter, working two sounders of different pitch—the one grave and the other acute.

Pellet's continuous analysis tube (Fig. 7).—A great delay in the analysis of beets occurs in the filling, emptying, and cleansing of the

saccharimeters, and the time occupied by those making the readings in inserting the tubes in the instrument and taking them out again. To economise time and labour, Pellet devised a method by which the whole of the analyses can be done by means of a single tube.

For this purpose he adapted two branches to a narrow tube 40 centimetres long, one at each end, quite near the lenses. The saccharimetrical tube being placed in position, if the small rubber tube dips into a glass full of juice, and the air sucked out, at the other a syphon is formed and the glass empties itself, unless the tube be pinched to stop the current. The apparatus being arranged in this way, a glass containing the juice is placed underneath the small rubber tube. The extremity of the long tube is released, and the saccharimetrical tube is filled with juice. The reading is taken, an assistant places another glass in place of the first, fills the syphon, and

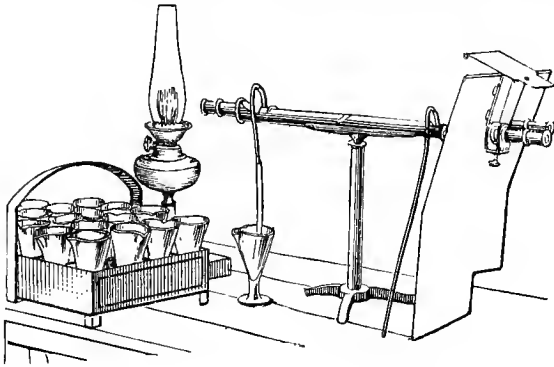


FIG. 7.—Apparatus for continuous saccharimetric analysis of beets.

the fresh liquid takes the place of the first. A new reading is taken, registered, and a third glass takes the place of the first, and so on. Ten thousand analyses may be done in this way daily by a staff of fifteen to seventeen persons.

Summary.—The estimation of the percentage of sugar in beets intended for seed has been brought to a high degree of perfection, combined with rapidity. In those large seed establishments, like Vilmorins in France, and Kleinvanzleben in Germany, which make the growing of beet seed a speciality, it was necessary to find rapid methods by which one operator could analyse thousands of samples a day. These methods have just been described, and it only now remains to insist here on the importance of the analysis of the beets and the immense number that have to be analysed in a very short time in the laboratories of beet seedsmen. These laboratories have been aptly termed analyses factories, with motor-power machinery,

and a large staff capable of working quickly and accurately; accuracy in that kind of work being as important as speed.

Hanriot's very elegant arrangement has been installed at the sugar factory of Logras et Cie. of Aulnois-sous-Laon, where 8500 analyses per day are made with a Leudeboom drill, a knife, and four Hanriot's apparatus, three of which are at work. In order to get the 93,613 beets necessary to produce the seeds used in laying down the crop of beet grown for this firm, they expended in 1897, according to Helot—

Chemicals	£83·58
Labour and supervision	69·132
Light	2·812
Paraffin oil for saccharimeter	0·7
Coal for engine	7·996
Loss and depreciation	6·23
	<hr/>
	£176·450

F. Sachs and A. Le Docte use an arrangement by means of which a considerable number of analyses may be got through in a comparatively short time. The German normal weight, 26·048 grammes, is weighed out into a suitable vessel, and by means of special automatic pipettes 5 c.c. of basic acetate of lead and 177 c.c. of water are poured on to it, the vessel covered with a suitable lid and well agitated. An exact measurement is thus obtained without bothering about the air contained in the pulp. All that remains to be done is to polarise in a 400-millimetre tube. Gallois and Dupont make similar pipettes for the French normal weight.

In the analysis of beets in France it is necessary to take the French normal weight for 200 c.c. and polarise in a 200-millimetre tube, as digestion is not perfect or complete with the normal weight for 100 c.c.

According to Peterman, the amount of sugar in the beet, estimated by digestion in water, is on an average $\frac{2}{10}$ per cent. higher than by digestion in alcohol. 98 comparative determinations were made by the alcohol and water methods, and the mean by the water polarisation was 12·98 per cent. Careful determinations were also made in comparing the alcohol extraction with the cold-water digestion on a very finely divided beet pulp according to the Pellet method. The means of 28 determinations were as follows:—Alcohol extraction, 12·72 per cent.; Pellet's cold-water diffusion, 12·73 per cent.; ordinary polarisation in water, 12·81 per cent. It is recommended that alcohol extraction be used in all cases where scientific exactness is required.

Preparation of basic acetate of lead solution.—Digest at a gentle heat 300 grammes of lead acetate with 100 grammes of litharge in 500–600 c.c. of water until completely dissolved, make up to a litre, decant after complete deposition.

[TABLE XX.]

BASIC LEAD ACETATE CORRECTION TABLE 63

XX.—CORRECTION TABLE FOR INCREASE OF VOLUME BY ADDITION OF BASIC ACETATE OF LEAD.

Degrees Read.	Corrected Degrees.	Percentage of Sugar Calculated on Volume.	Density of Solution.	Percentage of Sugar by Weight.	Degrees Read.	Corrected Degrees.	Percentage of Sugar Calculated on Volume.	Density of Solution.	Percentage of Sugar by Weight.
8	8·8	6·6	1·0255	6·44	16	17·60	13·20	1·0509	12·56
8·25	9·07	6·8	1·0263	6·63	16·25	17·87	13·40	1·0517	12·74
8·50	9·35	7·01	1·0271	6·83	16·50	18·15	13·61	1·0524	12·93
8·75	9·62	7·22	1·0279	7·02	16·75	18·42	13·82	1·0533	13·12
9	9·90	7·43	1·0287	7·22	17	18·70	14·03	1·0541	13·31
9·25	10·17	7·63	1·0295	7·41	17·25	18·97	14·23	1·0548	13·49
9·50	10·45	7·84	1·0303	7·61	17·50	19·25	14·44	1·0556	13·68
9·75	10·72	8·04	1·0311	7·80	17·75	19·52	14·64	1·0564	13·86
10	11·00	8·25	1·0319	7·99	18	19·80	14·85	1·0572	14·04
10·25	11·27	8·45	1·0326	8·18	18·25	20·07	15·05	1·0580	14·23
10·50	11·55	8·66	1·0335	8·38	18·50	20·35	15·26	1·0588	14·41
10·75	11·82	8·87	1·0343	8·58	18·75	20·62	15·47	1·0596	14·60
11	12·10	9·08	1·0351	8·77	19	20·90	15·68	1·0604	14·79
11·25	12·37	9·28	1·0358	8·96	19·25	21·17	15·88	1·0611	14·97
11·50	12·65	9·49	1·0366	9·15	19·50	21·45	16·09	1·0619	15·15
11·75	12·92	9·69	1·0374	9·34	19·75	21·72	16·29	1·0627	15·33
12	13·20	9·90	1·0382	9·54	20	22·00	16·50	1·0635	15·51
12·25	13·47	10·10	1·0390	9·72	20·25	22·27	16·70	1·0643	15·69
12·50	13·75	10·31	1·0398	9·92	20·50	22·55	16·91	1·0651	15·88
12·75	14·02	10·52	1·0406	10·11	20·75	22·82	17·12	1·0660	16·06
13	14·30	10·73	1·0414	10·30	21	23·10	17·33	1·0667	16·24
13·25	14·57	10·93	1·0422	10·49	21·25	23·37	17·53	1·0674	16·42
13·50	14·85	11·14	1·0431	10·68	21·50	23·65	17·74	1·0682	16·61
13·75	15·12	11·34	1·0438	10·86	21·75	23·92	17·94	1·0690	16·78
14	15·40	11·55	1·0445	11·06	22	24·20	18·15	1·0698	16·97
14·25	15·67	11·75	1·0453	11·24	22·25	24·47	18·35	1·0706	17·14
14·50	15·95	11·96	1·0461	11·43	22·50	24·75	18·56	1·0714	17·32
14·75	16·22	12·17	1·0469	11·62	22·75	25·02	18·77	1·0722	17·51
15	16·50	12·38	1·0477	11·82	23	25·30	18·98	1·0729	17·69
15·25	16·77	12·58	1·0485	11·99	23·25	25·57	19·18	1·0738	17·86
15·50	17·05	12·79	1·0493	12·19	23·50	25·85	19·39	1·0746	18·04
15·75	17·32	12·99	1·0501	12·37	23·75	26·12	19·59	1·0753	18·22

Use of the above table :—To 100 c.c. of juice 10 c.c. of basic acetate of lead are added and filtered. Suppose the rotation imparted to the plane of polarisation by a column of 20 centimetres be 18°, the table gives for the corrected degree 19·8; that is the deviation that would be obtained by using a 22-centimetre tube. 100 c.c. of the juice contain 14·85 grammes of sugar, or 100 grammes of juice contain 14·04 grammes.

XXI.—TABLE FOR THE ANALYSIS OF SUGAR BEETS. FRENCH SACCHARIMETER (F. SACHS).

Sacchari- metrical Degrees.	Sugar Per Cent. By Weight.		Sacchari- metrical Degrees.	Sugar Per Cent. By Weight.	
	In Juice.	In Beets.		In Juice.	In Beets.
55	9·29	8·83	88	14·51	13·78
56	9·45	8·98	89	14·67	13·93
57	9·61	9·13	90	14·82	14·08
58	9·77	9·29	91	14·98	14·23
59	9·93	9·43	92	15·13	14·37
60	10·08	9·56	93	15·29	14·53
61	10·24	9·73	94	15·45	14·68
62	10·40	9·88	95	15·60	14·82
63	10·56	10·03	96	15·75	14·96
64	10·72	10·18	97	15·91	15·11
65	10·87	10·32	98	16·06	15·26
66	11·03	10·48	99	16·21	15·40
67	11·19	10·63	100	16·36	15·54
68	11·35	10·78	101	16·51	15·68
69	11·51	10·93	102	16·66	15·83
70	11·66	11·08	103	16·81	15·97
71	11·82	11·23	104	16·96	16·11
72	11·98	11·38	105	17·12	16·26
73	12·14	11·53	106	17·27	16·41
74	12·30	11·68	107	17·42	16·55
75	12·45	11·83	108	17·57	16·69
76	12·61	11·98	109	17·72	16·83
77	12·77	12·13	110	17·87	16·98
78	12·93	12·28			
79	13·09	12·43	0·1	0·01	
80	13·24	12·58	0·2	0·03	
81	13·40	12·73	0·3	0·04	
82	13·56	12·88	0·4	0·06	
83	13·78	13·03	0·5	0·07	Same.
84	13·88	13·19	0·6	0·09	
85	14·03	13·33	0·7	0·10	
86	14·19	13·48	0·8	0·12	
87	14·35	13·63	0·9	0·13	

N.B.—This table is calculated for a normal weight of 16·29 grammes, in supposing that the average purity increases with the richness of the beet. The determination of the density is useless. 10 c.c. of basic acetate of lead must be added, for which 1 c.c. has been deducted for the space occupied by the plumbic precipitate. The observation is made in a 200-millimetre tube. A coefficient of 95 has been allowed for the beets, although this coefficient may be too high for rich beets.

XXII.—TABLE FOR THE ANALYSIS OF SUGAR BEETS. GERMAN SACCHARIMETER (F. SACHS).

Sacchari- metrical Degrees.	Sugar Per Cent. By Weight.		Sacchari- metrical Degrees.	Sugar Per Cent. By Weight.	
	In Juice.	In Beets.		In Juice.	In Beets.
30	8·15	7·64	56	14·82	14·08
31	8·40	7·98	57	15·07	14·32
32	8·66	8·23	58	15·31	14·55
33	8·92	8·48	59	15·56	14·79
34	9·19	8·73	60	15·81	15·02
35	9·46	8·98	61	16·06	15·26
36	9·72	9·24	62	16·30	15·49
37	9·98	9·48	63	16·55	15·72
38	10·24	9·73	64	16·79	15·95
39	10·50	9·97	65	17·04	16·19
40	10·76	10·22	66	17·28	16·42
41	11·02	10·47	67	17·53	16·65
42	11·27	10·71	68	17·77	16·88
43	11·53	10·95	69	18·02	17·12
44	11·79	11·20	70	18·26	17·35
45	12·04	11·44			
46	12·30	11·68	0·1	0·02	
47	12·55	11·92	0·2	0·05	
48	12·80	12·16	0·3	0·07	
49	13·06	12·41	0·4	0·10	
50	13·31	12·65	0·5	0·12	
51	13·56	12·89	0·6	0·15	
52	13·82	13·13	0·7	0·17	
53	14·07	13·37	0·8	0·20	
54	14·32	13·60	0·9	0·22	
55	14·57	13·84			

N.B.—This table is calculated for a normal weight of 26·048 grammes, in supposing that the average purity increases with the richness of the beet. The determination of the density is useless. 10 c.c. of basic acetate of lead must be added per 100 c.c. of juice, for which 1 c.c. has been deducted for the volume of the plumbic precipitate. The observation is made in a 200-millimetre tube. 95 has been taken for the coefficient for beetroot, although this figure may be in reality too high for rich beets.

The determination of the percentage of sugar in beetroot was for a long time made by *Violette's* process, based on the fact that when the beetroot pulp was treated by a dilute acid only the cane sugar is transformed into reducing sugars, which are titrated with copper solutions. By means of a small drill a cylindrical sample is taken from the upper third of the root, in such a way as to get a fair average

sample of the beet for analysis. The skin is removed and the small cylinder is cut into fine slices in the direction of its length. These slices are cut into finer slices and 10 grammes introduced into a graduated flask of 100 c.c., with 10 c.c. of normal sulphuric acid and 40 c.c. of distilled water, and the whole brought to the boil. After boiling for a quarter of an hour the solution is cooled, made up to 100 c.c., and titrated with Violette's solution. This process is at the present time, according to *Malpeaux*, almost abandoned. The operations, he says, are tedious, the end of the reaction difficult to catch, and the results obtained generally too high. On the other hand, *Ville*, no mean authority, stated that the estimation of sugars by means of the cupro-potassic solution is susceptible of very great precision. This precision, in his opinion, was at least equal to that given by the optical saccharimeter. In 1861 the two methods were the object of very careful comparison in *Ville's* laboratory on rather a large number of beetroot juices.

The following were the results obtained :—

XXIII.—ANALYSIS OF BEET JUICE. COMPARISON BETWEEN OPTICAL AND CHEMICAL METHODS.

	Chemical Process. Cane Sugar Per Cent.	Saccharimeter. Cane Sugar Per Cent.
1	7·31	7·47
2	8·49	8·63
3	7·37	7·74
4	7·94	8·12
5	8·67	8·70
6	9·71	9·71

CHAPTER III

PRELIMINARY TREATMENT OF THE BEETS—CONVEYANCE TO THE FACTORY—HEADING AND TAILING — WASHING AND CLEANSING — DRYING AND WEIGHING

Mode of conveyance to the factory.—The beets are transferred from the factory yard, where they have been stowed for the time being, to the factory proper, where the first operation to which they are subjected is that of *washing*. Several different methods of transfer may be adopted, each of them more or less economical, and more or less adapted to meet the special circumstances of individual cases. The best method of transfer is, of course, that which is the cheapest, quickest, and entails the least amount of handling and labour.

Transfer by barrows from silo to factory.—*Elimination of mechanical impurities.*—Shifting the beets by barrow is not practicable unless the heaps or silos are close at hand. Moreover, in any case the beets must be piled with care, so as to remove all stones, straw, etc., which might cause injurious complications. Accordingly, to eliminate these impurities, use is made of wide pronged forks which can only pick up the beets themselves. Sometimes, indeed, the labourers are made to take them from the heap by hand. This handling process is, of course, more laborious, but it is often a profitable one, when the roots are very dirty and liable to cause a stoppage in the factory by the breakdown of machinery.

Transfer by trucks running on tramway.—When the silos are at some distance away from the factory, small trucks running on light rails may be used. These rails are capable of being easily shifted, so as to run along the open silos.

Hodgson's cable system of transport.—Again, in certain cases where the depôt is very far off, cable transport is adopted. This consists essentially of a long endless steel-wire cable, stretching between the factory and the depôt, and driven by a steam-engine. The cable passes over a horizontal pulley at both ends, and thus forms two parallel ropes, travelling in different directions. Supports, in the shape of brackets, keep the cable at a sufficient height above the ground. In this way it can pass over roads, rivers, hill and dale,

throughout a course of several miles. The trucks, full of beets, are hooked on to this cable, and thus proceed, quite alone, to the factory, and are then returned empty from the factory to the depôt on the opposite rope. This system of transport is very useful in districts with bad means of communication; moreover, the cost of freight is relatively cheap, but dearer than by locomotive.

Feeding the washing machines.—The most economical method of feeding the beet-washing machines, from every point of view, is undoubtedly that known as the hydraulic carrier. It consists of a narrow channel or mill-lade of about 20 inches wide, rounded at the bottom, running right through the factory yard, in which a stream of water flows rapidly. When the beets are pitched into this stream, as their density is only slightly greater than water, they are carried along with the current to the end of the channel, which dips into the washer. There is not the slightest fear of a barrow-load of beets, thrown in at one point, stopping the flow of water, because at that point the level of the liquid rises, passes over the heap of beets, carrying away the top ones in its train, disintegrates the heap, and, under the ever-increasing pressure from behind, forces the individual beets to swim rapidly as far as the washer.

The yard may be radiated with or intersected by similar channels, and the labour connected with the handling of the beets is much diminished. Moreover, if the different channels be covered with boards, which cover them up completely, the heaps may be piled above the channel itself, and by lifting the boards in rotation a single labourer can dismantle them from the one end of the heap to the other, and thus feed the washing machine at the same time.

Water supply for washing the beets.—The hydraulic carrier is therefore the most convenient and cheapest method of transport when there is a slope towards the factory. The water which feeds it is that used for the washing machinery, for which purpose there has to be a very abundant supply.

Whatever may be the method of transport adopted to transfer the beets to the factory, they, in the first place, have to pass through one or two washing machines, so as to free them from the mud with which they are encumbered—a very difficult operation indeed, especially when they have been lifted in wet weather on heavy soils.

The different forms of washing machines.—*The old type.*—The washing machines formerly in vogue consisted simply of a wrought-iron drum, pierced with numerous holes of about three-quarters of an inch in diameter, revolving on a horizontal axis, beaters in the interior; and an exit for the wash-water, at the opposite end to that of the entrance, caused the beet to circulate from the one end to the other. This drum dipped to half its depth into a trough filled with water, and thus effectually scoured the beets.

The new type.—*Fricourt's beet-washing machine.*—At the present

day the cylinder has been superseded. Wooden blades fixed into an axis dipping into the trough have been found to meet all requirements. The blades are arranged in a helical manner, and force the beets to advance by turning them round and round until they make their exit from the tank, pressed against the sides by the helical motion of the blades. The diameter of these machines varies between 5 and 6 feet, and they are about 20 feet in length. Two of them always, and sometimes three, are arranged end-on to each other, the last being fed with fresh clean water to completely and thoroughly cleanse the beets.

The stone remover.—As the beets come from the last washer they pass through a small washer with iron blades in a deep tank, in which the water moves at a rapid rate. This washer is called the stone remover, because if any stones be carried forward by the

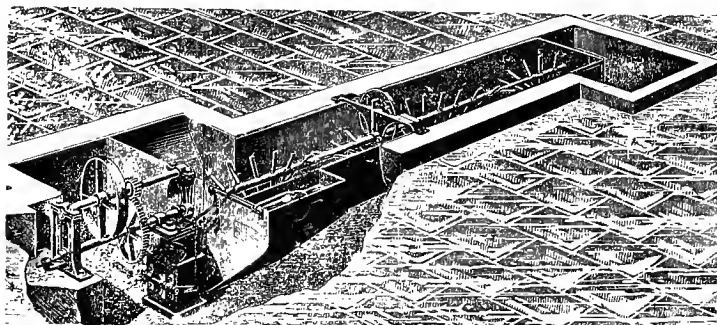


FIG. 8.—Fricourt's machinery for washing and cleansing beets and freeing them from stones.

washers they fall to the bottom of the beck without the blades being able to discharge them, as they do not touch the bottom, the water unaided bringing the beet to the surface. The stone remover is an essential machine in all sugar-beet factories, but more especially in the case of those which are fed with beets from pebbly clay soils. It might almost be dispensed with in the case of beets grown on soil free from stones. In the illustration of Fricourt's washing machine is to be seen an arrangement in the front—a sort of door driven automatically by gearing, which rises and falls alternately to and from a regulated height, so as to clear out any mud accumulated in the bottom.

Loze's stone eliminator for washing and cleansing sugar beets.—This machine is generally placed behind the rinsing washer. It may, however, be fixed in another position, if the existing arrangements require this to be done. It acts very simply. The machine is filled

with water and started. The helix, placed in a short cylinder, produces a rapid current of water, which passes through a grating on which the beets fall. The latter, carried up by the current, fall on to a second inclined grating, and from there on to a small paddle

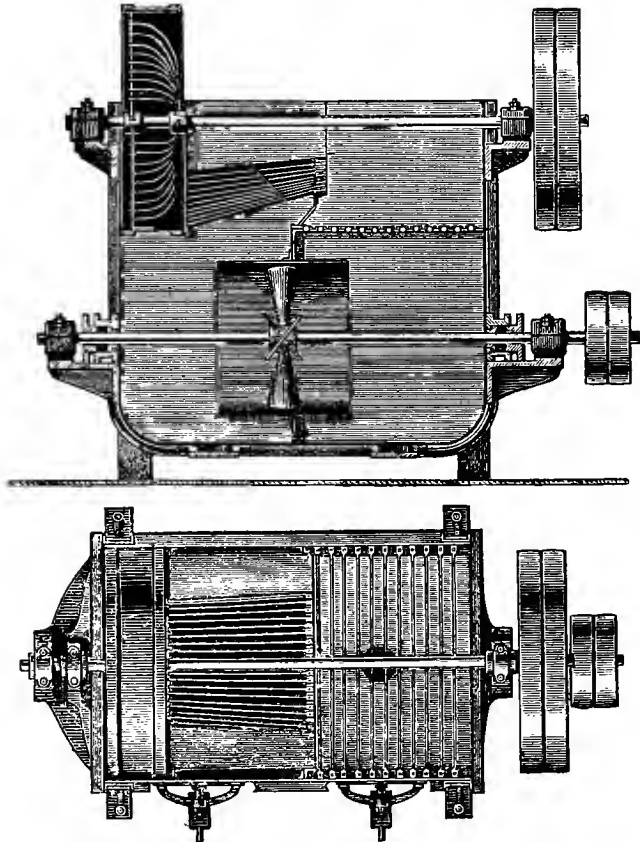


FIG. 9.—Stone eliminator, for washing and cleansing sugar beets (LOZE).

wheel, which throws them outside into the helix elevator or the succeeding washer. The stones or other small-sized heavy bodies fall through the grating into the bottom of the machine. The large stones or other heavy bodies remain on the first grating. The machine is emptied during stoppages (meal hours). The machine requires but little water, because the latter is not renewed except at

stopping-time. This machine may also be incorporated in the washing machine itself.

The beet, after being washed in the manner indicated, is then quite clean, and the next step is to extract the juice from it. Here the fiscal or excise laws of different countries come into play, and may slightly alter the extraction plant in the countries in question.

WEIGHING THE WASHED BEETS

A necessary process in France and other countries where sugar duty is levied on weight of beets.—When the tax is levied on the finished product, *i.e.* upon the sugar despatched from the factory, the beet is treated for its sugar immediately it comes from the washing machines. When the tax is levied on the weight of the raw material, treated as in France, the beets have to be weighed under the supervision of the Excise as they come from the washing machines before being treated.

The more complicated case—that of the weighing of the washed beets by the Excise—has the greatest claim on our attention, the more especially as it is an operation which is of such importance to the manufacturer that he ought to perform it himself, when the Excise do not insist upon it, so as not only to be able to know exactly the weight of the beets treated, but also to be able to check his stocktaking of the sugar production, and ascertain whether there is any loss in working, any preventible leakage, etc. The washed beets are therefore sometimes weighed in well-regulated and well-managed factories, where the tax is levied in some other way than on the weight of the raw material.

Drying the washed beets before weighing.—*Maguin's shaker.*—The beets as they come from the washing machines are wet. They therefore require to be dried, so as not to weigh too much water along with them, and thus pay duty on water as well as on the beets themselves. With this end in view, the beets are passed on to a shaker. This consists of a metallic sieve, in the form of a very slightly inclined plane, which allows the water to drain off. It is animated by a to-and-fro movement, which gives it a shake at each change of direction both fore and aft. This shaker (Fig. 11) is about 3 feet wide and 16 to 20 feet long, according to the size of the factory. It propels the beets to the hopper of the weighing machine at such a gradual pace that, turned round about and round about by each shake, they are sufficiently dry before they reach the end of their journey.

Denis Lefevre's machine for drying washed beets.—Attempts have been made to produce more perfect drying machines than those on the shaking-sieve principle. Thus Denis Lefevre's apparatus (Fig. 10) consists of a trough, the bottom of which consists of a series of parallel

revolving brushes, close to one another, their axes being perpendicular to that of the trough. These brushes, which are made of whalebone,

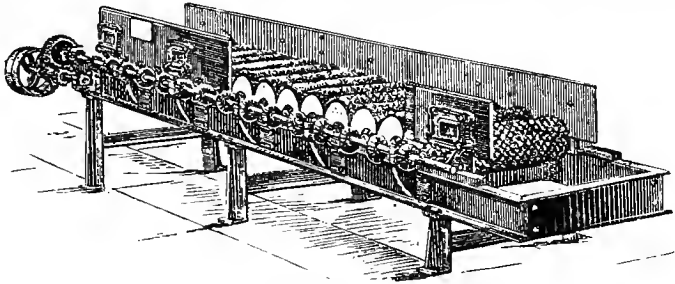


FIG. 10.—Beet-wiping machine (LEFEVRE).

propel the beets to one another, rolling them in every direction. The beets are therefore wiped more dry than they are by the shaker, only

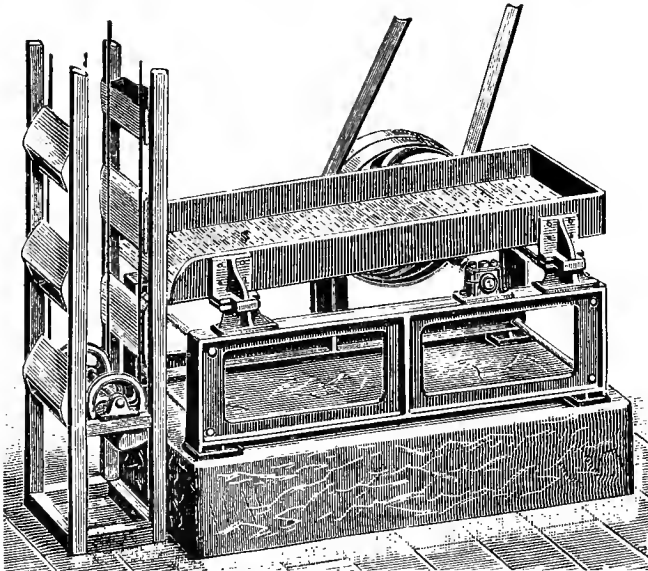


FIG. 11.—Shaker used in drying washed beets before weighing (MAGUIN).

the upkeep of the brushes entails a certain expenditure, compensated, moreover, by the smaller amount of water which passes to the weighing machines.

Hot-air drying ovens.—Attempts have also been made to dry beets in hot-air drying ovens. But no results of practical application have been attained. In any case, the beets pass from the drying machine to the weighing machine.

Elevators.—The beets are transferred from the carriers to the washing machines, from the washing machines to the drying machines, from the drying machines to the weighing machine (all appliances which cannot be fixed on the same floor), by means of elevators.

The different types of beet elevators.—*Paddle elevators, cup elevators, chain elevators.*—There are several kinds of elevators. Some consist simply of hemp or gutta-percha belts, inclined at an angle of 45°, on which paddles or cups are riveted or screwed. The beets, fed into the hopper at the bottom of the elevator, are raised to the desired height, at which point they are emptied and the string of empty cups descend to the bottom, to be again charged with beets at the hopper below.

Archimedean screw elevators.—There are other kinds of elevators, on the principle of the Archimedean screw, revolving in a frame having the same slope as the paddle elevator. The beet is in this case seized in the thread or path of the screw, and is raised up with the greatest facility.

Vertical chain and cup elevators.—But the two last classes of elevators cannot be made of any length on account of their slope, which makes them cumbersome. They are only used to raise beets a few feet. When it is required to raise beets to the top of the factory, where the weighing is usually done, vertical elevators (Fig. 12) are used. These consist of sheet-iron cups, attached at intervals to two parallel chains in place of a belt. The cups are sometimes a constituent part of the chains, held together by short chains which hook on to rings on the cups. In fact, the chain elevator has been still further simplified by inserting only a single chain between each cup. The latter glide, on each side, on a kind of wrought-iron rail screwed to the uprights of the elevator.

Whatever sort of vertical elevator be adopted, the beetroots fall directly into the cups below, which carry them to the top of the elevator, where they are shot down an inclined plane which leads to the weighing machine.

The requirements of the French Excise in regard to weighing machines.—The weighing of the beets must be done very accurately when the Excise is in charge, for neither the fiscal authorities nor the manufacturer want their respective interests to suffer. The French Excise authorities have accordingly imposed a series of very severe conditions and restrictions in the construction of the weighing machines in question.

The first condition is that the scoop of the weighing machine cannot be emptied until the exciseman has satisfied himself that the

weight is exact; and, secondly, that the exciseman himself cannot authorise its discharge until the balance is quite in equilibrium.

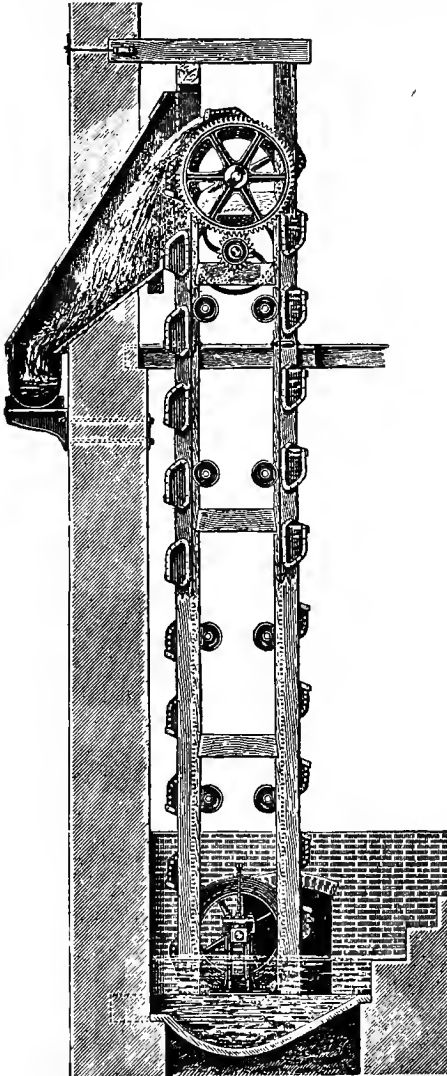


FIG. 12.—Vertical elevator for raising beets (MAGUIN).

Again, it is necessary that it should be impossible for the manufacturer to add a single beet after the weighing has been accomplished.

All these requirements have been complied with by numerous makers of weighing machines, in a rather simple and almost identical manner in all cases.

Briefly, a weighing machine (A, Figs. 17, 18, and 19), to meet the requirements of the French Excise authorities, consists of a scoop, with an inclined bottom placed on an ordinary steelyard. The scoop has a hinged lid above and underneath; on the vertical face opposite to the inclined plane of the bottom it also carries a hinged door, opening from below upwards. The two doors are connected by a system of cams and levers, which do not allow of one being opened until the other is closed; so that when the lid is raised the door below is bolted, and reciprocally the door underneath can only be opened when the lid above is bolted.

Owing to this, filling and emptying are quite distinct; not a single beet can enter the scoop before it has been completely discharged.

On the other hand, the framework of the steelyard, which supports the pans itself, carries an œillet intended to hold a bolt, and this bolt, which is fixed, cannot enter the œillet until it is quite opposite it, *i.e.* when the steelyard is in equilibrium and when the scoop holds the exact weight of beets, which is represented by the weights placed in the plate of the balance.

At the moment this occurs, the exciseman, who holds the lever of the aforesaid bolt, can push the latter into the œillet. But the lever during this movement acts upon another lever, which undoes the bolt of the lower door of the scoop and bolts the upper door, in such a way that, so long as the balance is not in equilibrium, it is impossible to open the lower door, and it is only when the exciseman has brought his steelyard into equilibrium that it is possible to empty the scoop. When the latter is discharged the exciseman restores the lever to its place, fixes the door underneath, and allows the upper door to be opened. But not only so; each time the exciseman pushes his lever this movement puts an automatic register in motion. Every time he opens the bottom door he puts a second automatic register in motion. Each time that the supervisor passes, these two registers should correspond exactly, with the exception of a false manœuvre, the details of which are registered in the books.

The system works without any great friction.—This method of weighing works well, and gives little trouble between the Excise and the manufacturer. Nevertheless, it is rather complicated; if anything gets out of order, it is a matter of regret for the factory. But this must be borne with, as it is necessary to collect the tax, says a well-known French writer; and the present tax on beets, in his opinion, is the most equitable of any.

German rules as to weighing not so strict as the French.—In Germany the tax on beet was formerly imposed in the same way as it

CHAPTER IV

DIFFUSION — BEET SLICERS — KNIVES AND KNIFE-HOLDERS — THE VARIOUS FORMS OF DIFFUSION — BATTERIES — THE CHEMICAL CONTROL OF DIFFUSION — THE UTILISATION OF EXHAUSTED PULP — RECENT DEVELOPMENTS — PULP ELIMINATORS — THE CHEMICAL CONTROL OF DIFFUSION

HISTORY.—*The early attempts of Mathieu de Dombasle.*—About the year 1830 Mathieu de Dombasle (1777–1843), a celebrated French agronomist, who not only in his day made improvements on many agricultural implements, but who was also one of the original creators of the beet-sugar industry, introduced a maceration process for extracting the juice of the sugar beet without pulping, rasping, or grating the beet, and without the use of hydraulic presses. The beets were cut into thin slices by a suitable rotary machine. The slices were then transferred to the first of a series of casks arranged in the form of a battery, the juice of the one being run on to the next. The beet slices were macerated in this first cask for about an hour with about their own bulk of water, at a temperature of about 212° F. After this treatment—the liquid having now acquired a density of 2° Baumé, sp. gr. 1.014—it was run off into a second similar cask containing fresh beet slices. From the second cask it was run into a third, and so on until it had passed through the fifth cask, charged in the same way, when, its density having reached 5½° Baumé, sp. gr. 1.040, it was suitable for defecation. The first cask was thus charged with hot water, and the last cask yielded a juice suitable for further treatment. So as to prevent the juice from cooling, it was reheated in the course of its passage through the casks, and the greatest possible maximum amount of sugar was therefore extracted in this way. The exhausted slices contained almost no more sugar. But the juice obtained in this manner, although transparent and requiring but little lime for its purification, was liable to ferment, or, owing to the dilution water, it was difficult to granulate. Hence the process was generally abandoned, as the juice obtained by it could not be successfully treated by the method then in vogue for pressed juice, and because

the exhausted slices were so wet that they could not be used for cattle-feeding purposes.

The double carbonatation process clears the way.—And so matters remained for some twenty years, when, in 1849, Rousseau made a great step in advance by indicating a practical process of working beet juice, by which juice, of no matter what nature or how obtained, could be successfully treated.

In 1849 *Perier* and *Possoz* put the finishing touches to *Rousseau's* process, and their system of treating the juice was termed the *double-carbonatation* process. About the same time *Jelinek*, in Austria, adopted an almost identical process for treating the juice.

Final triumph and economy of the diffusion process.—Owing to the progress then made, it was found practicable to treat the maceration juices obtained according to the method of *Mathieu de Dombasle*. Moreover, about 1860 this process was again brought to the fore by the efforts of *Robert*, a sugar manufacturer of *Seelowitz*, Austria, who, out of the rudimentary appliances of *Mathieu de Dombasle*, evolved quite a new industrial system of treating the juice, called the *Diffusion Process*—a process which was destined to revolutionise the whole beet-sugar industry. He not only made *de Dombasle's* process practicable, but even economical. Table XXIV. shows how extraction by pressure was eventually totally superseded by diffusion.

The result of the action and reaction of the different improvements in the treatment of the juice.—The diffusion process therefore, says *Horsin-Deon*, only became practicable after the methods of working had been improved. Again, these methods could not have themselves been improved had it not been as a result of the invention of new machines. It has therefore been well said that the majority of innovations in sugar manufacture are connected together by a common bond. As chemists of high standing, engineers and manufacturers of great merit, have each and all tried all the improvements which their fertile brains could suggest, it is rare to find a real novelty in recent inventions. But those inventions, which were formerly impracticable, have only come into use in virtue of the immense progress in engineering in virtue of improved plant. Nevertheless, continues *Horsin-Deon*, each invention which sees the light of day brings its seal of indisputable novelty to the cause even of that progress from which it flows, even although it be only an old make-new. In this way was diffusion born and developed in our own days, thirty years after it was forgotten. In this way we have likewise seen carbonatation regenerated, after the invention of filter presses twenty-five years after *Kuhlmann* first thought of it; and *Steffen's* process of cleansing cuites sixty years at least after the American *tigers*. Had our fathers known what we know to-day it is evident that we would have nothing further to invent; and who knows what practical use our sons may

make of our modern researches, often without practical application? Happy are they who know how to profit by the knowledge of their predecessors. They are few and far between. But jealous incapables, pirates, and litigants, they are legion. This is what explains the numerous actions of nullity of patent, which have stopped somewhat the inventions of our time; for why should one work if the laws insufficiently protect those who spend their intelligence and their money in the cause of progress? There have been diffusion lawsuits, carbonatation lawsuits, filter-press lawsuits, evaporation lawsuits—lawsuits against everything. It is history; and it may be beneficial to mention in passing, in a résumé like the present, the difficulties in the genesis of the sugar industry. May our successors be more fortunate (Horsin-Deon).

Horsin-Deon's contentions, as given in the above paragraph, are no doubt true and to the point; but there are numerous other industries in regard to which a similar view might be taken as to both past and future progress, and there are but few discoveries in any branch of science or industry which do not dovetail into, merge in, or depend upon, some previous discovery. As to patent lawsuits, nullity, infringement, etc., each industry has had its own share—*e.g.* the Torbanehill mineral case, involving a scientific definition of the word "coal."

The scientific principles on which diffusion is based.—Although at first sight apparently rather complex, the principles underlying the process of diffusion are, as a matter of fact, very simple. When a well-cut thin slice of beet comes in contact with water an exchange of liquids is made, between the saccharine juice contained in the beet cells and the water in contact with the slice of beet, according to what is called the laws of osmosis or dialysis.

Osmosis and dialysis.—If we run in a solution of common salt in water into a bladder and tie it up securely, and suspend the bladder in a vessel containing distilled water, we will find after a certain time that the water outside the bladder is as salt as that inside the bladder. Equilibrium tends to establish itself, and at the end of a certain time—of greater or less duration—the two liquids acquire the same degree of concentration. Now, if instead of a solution of salt we take a weak solution of glue—so weak, in fact, that it cannot set into a jelly—we then find that, as a matter of fact, practically none of the glue permeates through the bladder into the water in the outer vessel. Again, if a mixture of solutions of glue and salt be run into the bladder and similarly treated, the two substances will be found after a time to have separated, the salt having passed through the bladder into the outer vessel, and the glue remaining behind inside the bladder. A great number of substances, especially those capable of assuming the crystalline form, behave in a similar manner to salt, and pass through the bladder—*e.g.* sugar. This can be demonstrated in a

somewhat similar manner to our experiment with salt. If we take a piece of rather wide glass tubing, and cover it closely and securely over at the end with bladder, and fill it with a solution of sugar, and then close it at the other extremity with bladder in the same way, and immerse the tube in distilled water, the bladder closing the ends of the tube will, after a certain lapse of time, be found to be distended or bulged outwards, owing to the pressure of the water which has been gradually absorbed from without inwards. The water in the outside vessel will at the same time have acquired a sweetish taste, showing that a portion of the sugar solution must have passed through the bladder from within outwards.

Osmosis differentiated into endosmosis and exosmosis.—This diffusion of fluids through a closed membrane is called *osmosis* (Greek, *osmos*, an impulse or pushing), the passage of the fluid from without inwards being differentiated as *endosmosis*, and the passage of the fluid from within outwards as *exosmosis*.

Crystalloids and colloids.—Again, substances the solutions of which pass through the animal membrane in the same way as salt and sugar are found to do are termed *crystalloids*, whilst those bodies like glue which do not pass through the membrane are termed *colloids*. This differentiation of substances into *crystalloids* and *colloids* is more relative than absolute, because *crystalloids* themselves pass through porous membranes at different speeds, and even *colloids* are not absolutely deprived of this property. It would be more correct to say that there is an ascending and descending scale of the osmotic properties of bodies, in which certain crystalline substances are placed at the top, whilst certain amorphous bodies occupy the bottom of the list.

Osmosis, therefore, is a physical process which goes on between two liquids separated by a permeable membrane.

Differentiation between osmosis and simple filtration.—But it must be borne in mind that a membrane may be permeable to osmotic phenomena without acting as a filter. Filtration and osmotic phenomena must not be confused. In the same way a membrane may be permeable to gases without being permeable to liquids. (Gases are subject to the same laws of osmosis as liquids.) Indiarubber, for instance, is permeable to gases, whilst it is quite impermeable to liquids, or at least to those which do not act upon it—*e.g.* children's balloons. Parchment paper, again, is permeable to liquids, but nevertheless cannot be used for filtering purposes. The same laws apply to the membrane of vegetable cells which are essentially favourable to osmosis, for osmosis is one of the vital principles of plant-life.

Summary.—When two liquids, therefore, of different densities are separated by a porous membrane there is intercommunication between them, and a passage of the two liquids respectively

through the membrane, in opposite directions, and the speed of the passage is inversely proportional to the density of the liquid,—that is to say, that the less dense liquid will pass through the quickest.

If one of the two liquids in solution be sugar, the saccharine fluid will pass through the membrane in one direction, whilst the pure water passes, in an inverse direction, much quicker than the saccharine fluid passes outwards. It follows, therefore, that the sugar content of the saccharine fluid in the beet cell becomes less and less, whilst that in the exterior fluid increases more and more, and the phenomena of diffusion, of course, ceases when the sugar content of the fluids is the same on each side of the membrane or diaphragm. Equilibrium has been established.

Now, if at this point the exterior saccharine fluid be run off and replaced by pure water, osmosis starts afresh, and by renewing the water indefinitely the sugar content of the original liquid inside the membrane is finally reduced to nil.

It will readily be conceived, therefore, that if a current of pure water be made to circulate in the outer compartment the whole of the sugar contained in the interior compartment will gradually pass through the partition. The cellulose, which forms the partition of vegetable cells, behaves like a permeable partition to *crystalloids*. On the other hand, it only allows gelatinous substances, *e.g.* albumenoid bodies, to traverse it with extreme difficulty. The principle, therefore, of the diffusion process of sugar extraction rests on the phenomena of osmosis. The actual operation is carried out by causing a current of water to circulate round the slices of beet until they are exhausted of all the sugar contained in their cells.

The diffusers, their general arrangement.—*The sugar-beet diffuser.*—A diffusion battery consists of a series of eight to fourteen cylindrical vessels arranged consecutively, and called *diffusers*. They communicate with each other by a system of pipes, in such a manner that the juice, issuing *from the bottom* of one diffuser, flows into the next *from above*. Moreover, by a system of taps the current may be reversed, if need be, so as to pass from top to bottom of the diffusers, instead of from bottom to top. But not only so, the juice may be heated during its passage from the one battery to the other.

The calorisor.—A steam reheater, technically called a *calorisor*, enables the liquid to be always kept in a hot state. Again, taps are also arranged so that water may be run into each diffuser, instead of juice. In addition there is a tap for running off the liquid after the beets have been exhausted.

Charging and discharging doors.—It is perhaps needless to mention that the diffusers have a door on the top for charging them with fresh beet slices, and another door at the bottom through which the exhausted beet slices are discharged.

Theory of the working of sugar-beet diffusion batteries.—Starting

with the supposition that all the diffusers are charged with fresh beet slices, there will still be a certain amount of vacant space between the slices. When a sufficient volume of water is run into the diffuser so as to occupy this vacant space, the diffuser will then

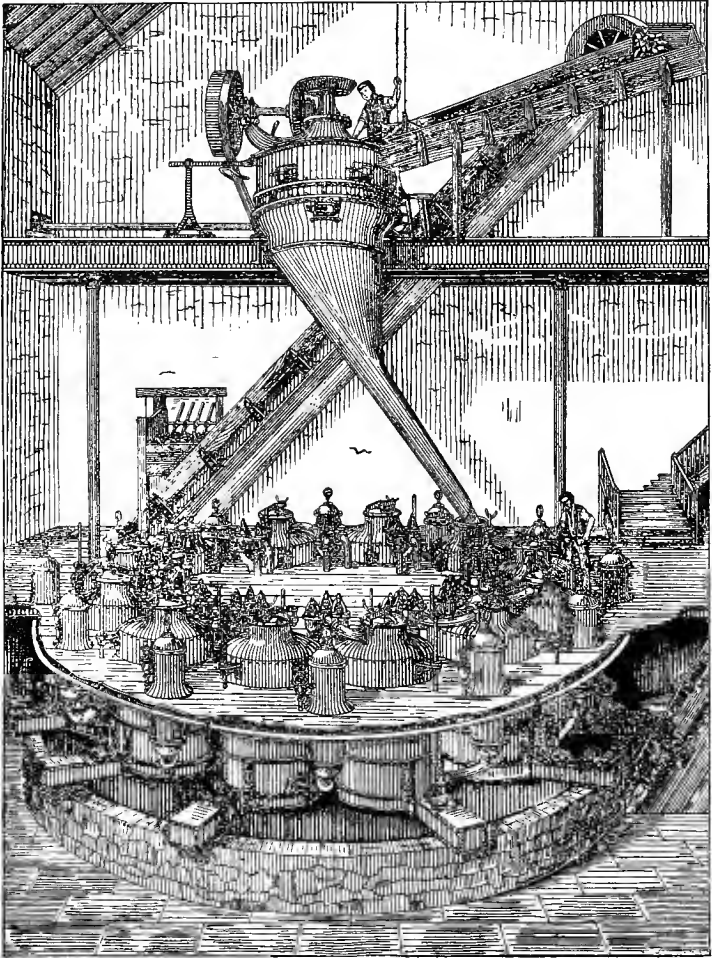


FIG. 13.—Circular diffusion battery for extracting the saccharine juice from beets by diffusion, showing inclined beet elevator, slicing machine, revolving funnel feeder, diffuser, stager, and pit into which exhausted slices fall.

contain about equal weights of water and beet slices. Not only so, but the volume of space occupied by each is also almost identical, the density of the beet being only slightly above that of the water.

If the first diffuser of the series be now charged with water, *osmosis* at once starts to act in the cells of the beet in such a way as to cause a certain proportion of the sugar which they contain to pass into the water.

The workman in charge of the battery, however, does not wait until equilibrium in density is established between the saccharine fluid contained in the cells and the exterior saccharine liquid, because that would take too much time. After a few minutes contact, however, there is no very great difference in density between the two. The liquid from the first diffuser, such as it is, is now run into the diffuser next to it in the series, it being reheated in its passage. The saccharine liquid is now in contact with fresh beet slices, whose juice is of greater density than its own, *osmosis* is again energetically started, consequently it now soon becomes still more highly charged with sugar, until, in fact, the densities are nearly equal. The saccharine liquid is once more run into a fresh diffuser, and the same interaction of fluids takes place, and the same operations are continued until the density of the liquid is only slightly inferior to that of the juice in the cells. The *osmotic* action is then almost nil.

The superior quality of diffusion juice.—Let us take it for granted that the passage of the liquid through the diffuser is stopped at No. 6 diffuser, after which it is sent to other departments of the factory for further treatment. The sugar content of the *diffusion juice*, as it is termed, is now so great that its density is approximately the same as the primitive juice of the beet. In addition to sugar, however, the beet also contains salts in solution, which have passed through the membrane of the beet cells simultaneously with the sugar. On the other hand,—and herein lies one of the many advantages of the diffusion process,—*the diffusion juice contains little or no colloids, such as vegetable albumen, pectose, etc.* Diffusion juice, consequently, is much more pure than real beet juice, for the reason that with both hydraulic and continuous presses the real beet juice is obtained by the more complete shredding of the cells by the teeth of the pulper or grater machine, so that it includes not only the crystalline sugar, but all the colloid bodies present in the juice.

It is therefore one point gained, one point in advance, to be able to demonstrate that diffusion juice is far superior, so far as purity is concerned, to that obtained by pressing the grated or rasped pulp.

The sequence of diffusion operations.—Having now obtained a clear idea of the way in which the sugar is extracted from each diffuser

in succession, and of the advantages such juice possesses over pressed juice, let us still further follow the sequence of operations. We left the diffusion battery in the following condition:—No. 1 contained half-exhausted beets; No. 2, quarter-exhausted; No. 3, one-eighth exhausted; and so on. If pure water be now run into No. 1, and circulated in the consecutive diffusers as before, No. 1 will be three-quarters exhausted, No. 2 will be half-exhausted, and so on, until we stop at No. 7 diffuser, by which time the diffusion juice thus obtained is quite as dense as that obtained in the first cycle of operations. Similar phenomena occur in the next and succeeding cycles: thus if water be once more run into No. 1 diffuser it will after that be seven-eighths exhausted, and this time the juice is circulated to No. 8 diffuser before running it off for treatment, and so on until the last diffuser of the battery is reached.

The diffusion extraction process a continuous one.—It will readily be understood that this continuous sequence of operations can be carried on uninterruptedly or simultaneously in such a way that the diffusers are always filled with liquid; as a matter of fact, that is the actual way in which operations are conducted, and the juice of the one diffuser pushes the juice of the other in front of it out of the one diffuser into the other. So as to accomplish this, water under pressure is run into the first diffuser, which drives in front of it the juice contained in that diffuser; the juice of the second diffuser displaces that of the third, and so on, until the last diffuser is reached, from which it escapes into a gauged reservoir, called the measuring tank, in which only that amount of juice which corresponds with the capacity of the diffuser is collected each time that the operation is repeated. From what has just been enunciated it will be readily understood that the sugar content of the juice as it is drawn off will be greater or less, and the exhaustion of the beet slices in No. 1 more or less perfect according to the volume drawn off and the interval between each repetition of the process.

Calorisators, their functions and action.—Further, as there is a calorisorator between each diffuser, and as increased temperature shortens the osmotic action and consequently facilitates exhaustion, care is taken to regulate the heat so as to maintain a temperature of 75° to 80° C. (167° to 176° F.) in the diffusers.

It will be readily seen that the greater the number of diffusers in operation the more effectual will be the exhaustion of the beets, as a greater quantity of pure water will have to be run into No. 1.

Comparative exhaustive effect of batteries with (a) a larger and (b) a smaller number of diffusers.—Of two batteries with a different number of diffusers, the battery with the larger number will do the greater amount of work, on account of the larger volume of water run into No. 1. The same result may be obtained by diminishing the time occupied by diffusion in each of the batteries. But, at the

same time, it would be necessary to heat a short battery to a greater temperature than a long battery, to the injury, moreover, of the quality of the juice, because *osmosis* is more energetic in the hot than in the cold; hence the colloid bodies dialyse more easily in the hot than in the cold, and consequently the juice is more impure.

The general routine of diffusion work.—Suppose all the elements, *i.e.* the diffusers of the battery, to be charged, and that No. 1 has to be emptied. This is done, and it is refilled with fresh beet slices. No. 2 diffuser now becomes No. 1. The next run, the new No. 1, is emptied, and the original No. 3 now becomes No. 1, and so on.

In order to work systematically and to economise time in the rather protracted operations of filling and emptying, matters are so arranged that in each battery there is always a diffuser being filled at the tail end and a diffuser being emptied in front, and it is the diffuser following the latter which is under hydraulic pressure.

Another important detail of the routine of working is that, when it comes to the last diffuser, instead of running in the juice from above downwards on to the fresh beet slices, it is run in from below upwards. The object of this manœuvre is to lift up the slices, and to mix them with the water or to mash them. Were the water to be run in from above, the slices would be piled in the bottom, and admixture of the beet slices and the juice would be imperfect. It is the juice of the last diffuser, after mashing, which is sent to the factory as the most concentrated, and it is the juice following which is used to mash the last diffuser.

Method of working diffusion process with short water supply.—In certain factories with a short supply of water, instead of pushing the juice of the last diffuser out by the pressure of the water reservoir or by hydraulic pressure, *compressed air* is used for the purpose. In this way three-quarters of the water which is generally lost in emptying the last diffuser is saved.

THE CONSTRUCTION OF DIFFUSERS, BATTERIES, AND THEIR ACCESSORIES—ROOT-SLICERS, KNIVES, KNIFE-HOLDERS, AND EXHAUSTED SLICE PRESSES

Beetroot slicers.—*The principles on which they should be constructed.*—The shape of the slices into which the beet ought to be cut before being run into the diffusers cannot be more aptly defined than by comparing it to the slices of vegetables used in making soup.

The success of the diffusion process depends on the size and shape of the slices.—The beet slice in this form should be regular, long, and well cut. These are the three important points. The *size* of the section varies with the nature of the beet. There is every advantage in cutting the slices as thin as possible—say one-fifth to one-sixth

of an inch square. Should the beet, on the other hand, be tender or softened by drying or frost, large-sized slices should be made. On the selection of the size of the slice often depends the successful working of the battery, and more especially the thorough exhaustion of the slices,—in other words, the quantity of sugar lost by the factory in the residues. As it is to the interest of all concerned to lose as little as possible of the sugar contained in the beet, the choice of the size of the slice is an important matter.

The *shape* of the slice has also a great influence on the yield; rectangular slices become glued together and obstruct the circulation of the water. By imparting to them the bent shape of the tiles used to cover the ridge of a roof, a large number of channels are created in the mass of slices which greatly facilitate the circulation of the water over the whole of the surface of the slice. These different shapes and sizes are obtained by imparting an appropriate shape to the root-slicers, and in arming them with good cutting knives acting at sufficient speed to make a neat cut.

In order to attain these different results, all efforts have been concentrated to impart to the root-slicers the most desirable shape.

Description of a typical beet-slicer.—*The revolving plate, the knives, and knife-holders.*—The essential organ of a beet-slicing machine is a circular horizontal plate revolving rapidly round a vertical axis. This plate is pierced with apertures in which knives are inserted. These are arranged like the cutting edge of a plane. It will be seen that if the beet comes in contact with this revolving plate the knives will plane the beet, and that the slices cut off the beet will fall beneath. Moreover, if the knives have an appropriate shape the shavings or slices cut off will have the desired shape which has just been described.

The feeding of the beets into the hopper and the way in which they are cut.—The revolving plate is enclosed in a frame surmounted by a hopper and shaped underneath like a large funnel. The beets are fed into the hopper, and, as the latter is of a certain height, 20 to 40 inches, the weight of the beets above, pressing on those below, and which rest immediately above the plate, acting as an automatic pusher, causes the latter to be caught by the revolving knives and thus to be cut into slices of the desired size and shape. The beet-slicer is therefore nothing more nor less than a special adaptation of the turnip-slicer so long extensively used in Britain for cattle-feeding purposes.

The fitting of the knives into the knife-holders and the knife-holders into the plate.—The apertures of the plate in which the blades are fixed are from eight to ten in number. The knives are not fixed directly into the plate. They are mounted in movable, easily changeable frames, called knife-holders, and it is these knife-holders which rest in the apertures of the plate.

Removing blunt knives and inserting a sharp set.—As the knives soon become used up, there is always a complete complementary set in reserve, which has been provided and adjusted beforehand, so that when the knives at work do not cut properly the knife-holders with the blunt blades are removed bodily, and immediately replaced by the previously prepared sets. The change is thus effected very rapidly, for the construction of the apertures is such that the change involves no difficult operation, the knife blades simply fitting into the grooves which secure it immovably during rotation. They are, in fact, kept in place by centrifugal force.

Gearing.—The plate is driven by conical gearing acting on its axis, the gearing being driven by belts and pulleys. The axis itself rests in a socket, and is kept in the vertical position by strong plunger-blocks surrounding it on the top in such a way that it cannot shift its position.

Diameter of the plates, and speed of revolution.—The diameter of the plates is very variable. The average is 5 feet. But some are 3 feet, and others over 6 feet, in diameter. Plates of great diameter are much in vogue in Austria. However, as the speed of the knives ought always to be the same, plates of large diameter must revolve more slowly than smaller ones. Plates of 5 feet in diameter make 100 to 120 turns, so that $6\frac{1}{2}$ -feet plates need only make 60 to 90 turns for their circumference to have travelled the same space in the same time as the 5-foot plates.

The different kinds of knives in use.—Knives may be divided into three classes—

1. *Naprawil knives.*—The first, the oldest, called Naprawil knives, make rectangular slices. They consist of a straight cutting blade surmounted at intervals by cutting ridges, which divide into small sections the slices cut off by the knife.

2. *Goller knives.*—The second class of knives, known as Goller knives, make triangular slices. They are steel blades $\frac{1}{8}$ to $\frac{1}{6}$ inch in thickness, cut in the body in a zigzag form at an angle of 60° . They are also made of wrought iron bent into an undulating form having the same profile. It will thus be seen that the beet can be cut into *triangular* slices. However, the form of the slice is quite irregular, because, when the knife passes through the beet it leaves its triangular mark upon it also. It follows, therefore, that when the next knife comes into play it cuts the ridges of the triangle, forming an irregular-shaped slice. This is bad, because the new slice has not the thickness requisite for good diffusion working, because the thin slices are exhausted sooner than the thick ones, and consequently the exhaustion is altogether imperfect and incomplete.

3. *The roof-ridge-tile-shaped knives.*—The third class of knives, the most extensively used, are the roof-ridge-tile shape. These combine the principles of the Goller and Naprawil knives. They

have the same profile as Goller's knives; only, on the upper part the summit of the angle carries cutting ridges like the Naprawil knives. It follows, therefore, that when a knife has passed, leaving the mark of its shape on the beet,—when the next arrives, if it passes into the same furrow, it lifts a roof-ridge-tile-shaped slice of perfect shape, and sharply cut on all its faces. By taking care to mount the knives in such a way that their blades are correctly placed one behind each

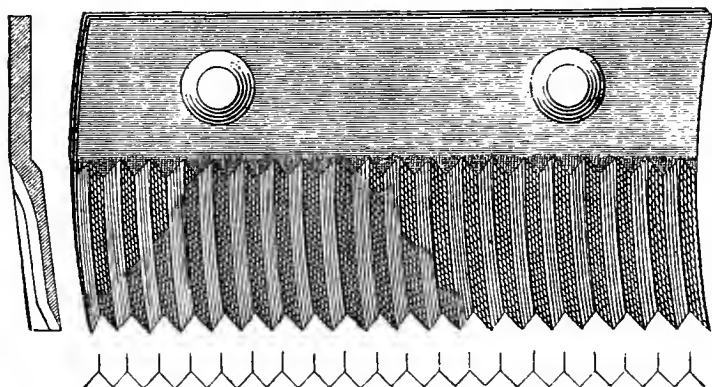


FIG. 14A.—Knives (MAGUIN).

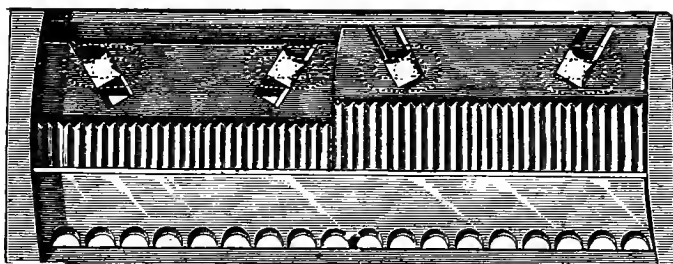


FIG. 14B.—Knife-holder, non-stone remover.

other, it is possible to get perfectly regular slices. Nevertheless, many slices are still defective, but less so than with the Goller knives.

Knife-holders.—The knives are mounted in a knife-holder like the chisel of a plane. So that the knife may catch more or less as the slice is to be larger or smaller, all that has to be done is to vary the slope of the knife and the length of steel which overlaps the plane of the knife-holder.

Method of sharpening and tempering the knives.—The knives are

sharpened on very hard steel discs which revolve rapidly on their axis, and the circumference of which is dressed like a file. The profile or shape of the bottom of the blade is given to the circumference, and it is enough to place the knife in front, firmly held in a clip which guides it, and to press lightly with the hand, to sharpen its cutting edge. There are also similar discs for sharpening the cutting edges of the ridges. In this way a knife is sharpened very quickly. The sharpening is finished by files also of the desired profile or shape.

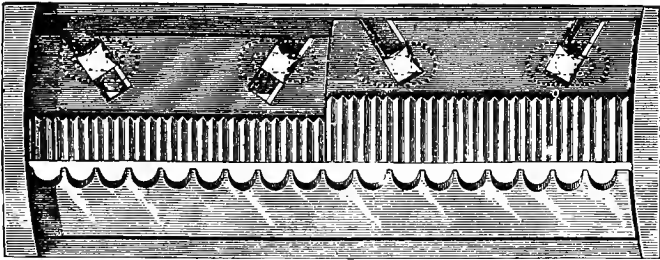


FIG. 14c.—Knife-holder and stone remover.

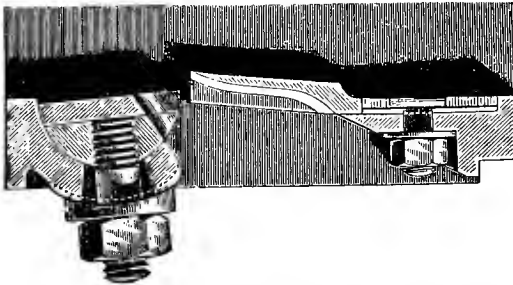


FIG. 14D.—Knives and knife-holders of the beet-slicers (MAGUIN).

Hardened untempered steel, and tempered steel knives.—Some knives are made of hardened non-tempered steel. These are sharpened immediately they are taken out of the beet-slicer. These are the class of knives most generally used. Some are made of tempered steel, and it is necessary to soften them before sharpening and to temper them afterwards. It is a big job, which requires great care to do it well, and consequently involves a special smithy and a skilful experienced smith, for which reason it is practised in but few factories.

The knives in current use only have been mentioned. There are

others derived from the three described, only different by unimportant details which need not be dwelt upon here.

Fontane and Rassman's beet-slicer.—Another form of root-slicer

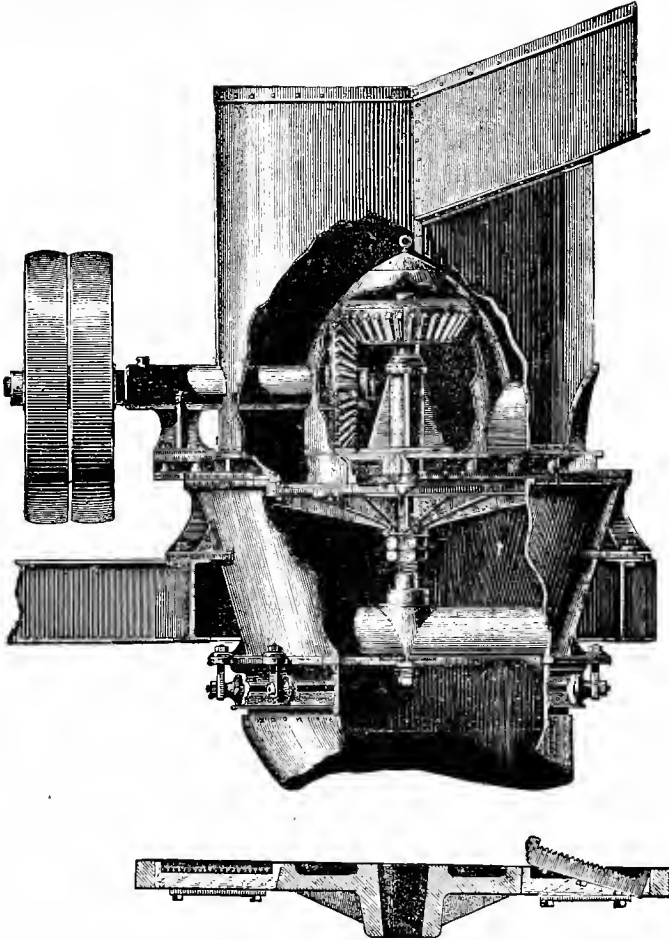


FIG 15.—Beet-slicer (FONTANE AND RASSMAN).

invented simultaneously by Fontane in France and Rassman in Germany, of quite a different shape, has had some success, without, however, having come into any extended use.

Description.—It consists of a vertical cylinder pierced on its

circumferences by eight to twelve apertures in which the knife-holders are fixed. The cylinder has the height of two superimposed knife-holders.

Method of working and the function of its parts.—In the axis of this cylinder a shaft turns very rapidly. This shaft carries two blades, which sweep the knives in turning.

Beets are run into the cylinder, where they are seized by the blades, and the centrifugal force presses them against the sides of

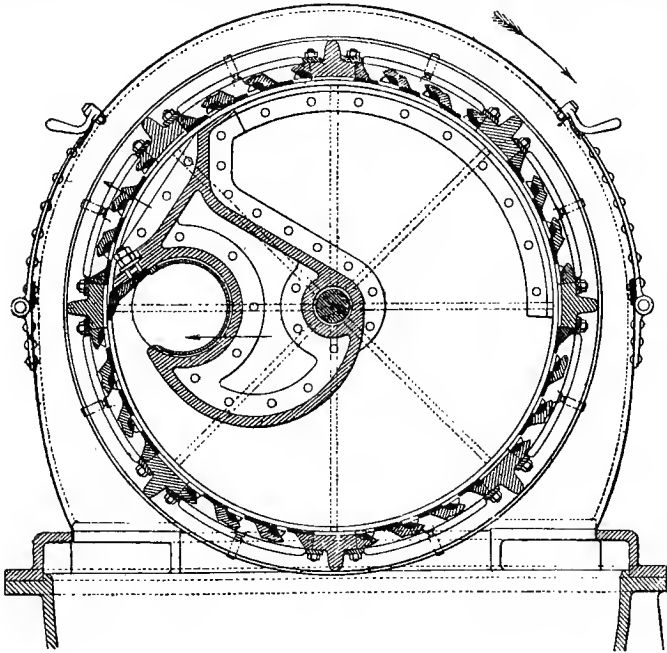


FIG. 16.—Rotary drum root-slicer (MAGUIN). Cross section.

the cylinder. Carried round by the blades, the roots are cut by the knives and thrown outside in very uniform slices.

The horizontal plate machine still preferred.—Other root-slicers on the same principles have since appeared, but the horizontal plate machine is still preferred by manufacturers. The following recent machine, however, well merits a special description :—

Maguin's rotary drum root-slicer.—In this machine all the knives are parallel. The knives revolve in every point at the same speed. They are close together, and the root is held until cut. These conditions are obtained by the following arrangements. The drum,

1.2 metres (47 inches) in diameter, .33 metre (13 inches) of interior cutting width, mounted on a horizontal axis, carries knife-holders to the number of eight all over its surface. Each knife-holder is furnished with six parallel knives, in pairs, with an instantaneous regulating plate in front of each row of two knives. These eight knife-holders therefore represent twenty-four ordinary knife-holders,

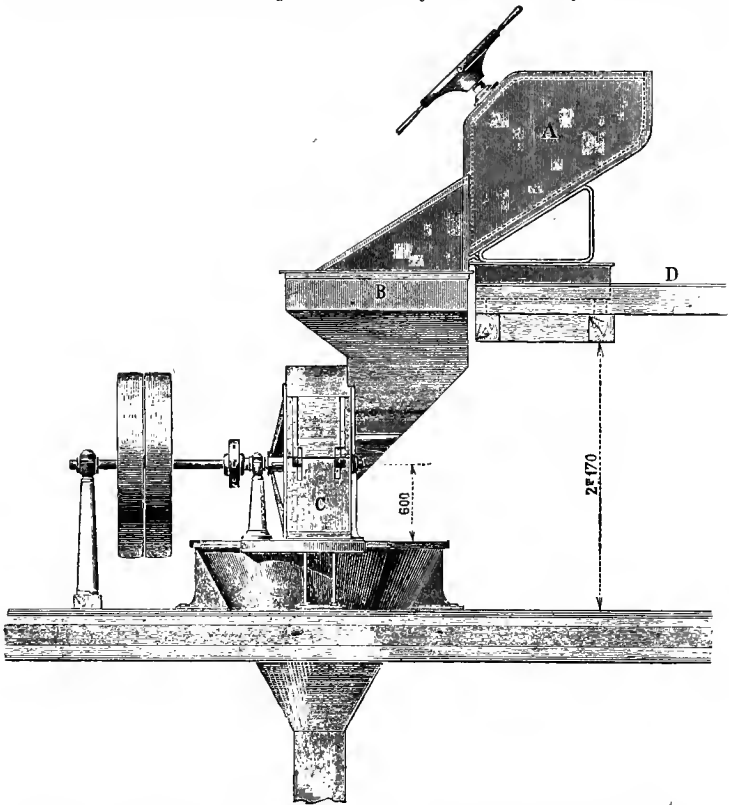


FIG. 17.—Rotary drum root-slicer mounted on body of old root-cutter (MAGUIN).

and carry forty-eight knives of 165 millimetres ($6\frac{1}{2}$ inches) in length in twenty-four parallel rows of two knives.

Being parallel, these twenty-four rows of knives go at the same speed. The beet, as soon as the half of its diameter is introduced, is carried by the circular part of the drum to a more and more restricted section formed by a rigid piece of cast iron in the form of a hook. The beet is carried in by the knives, which, being very close, slice it,

following the same edge, the more so as it is constantly kept in its position and pressed against the cutting surface.

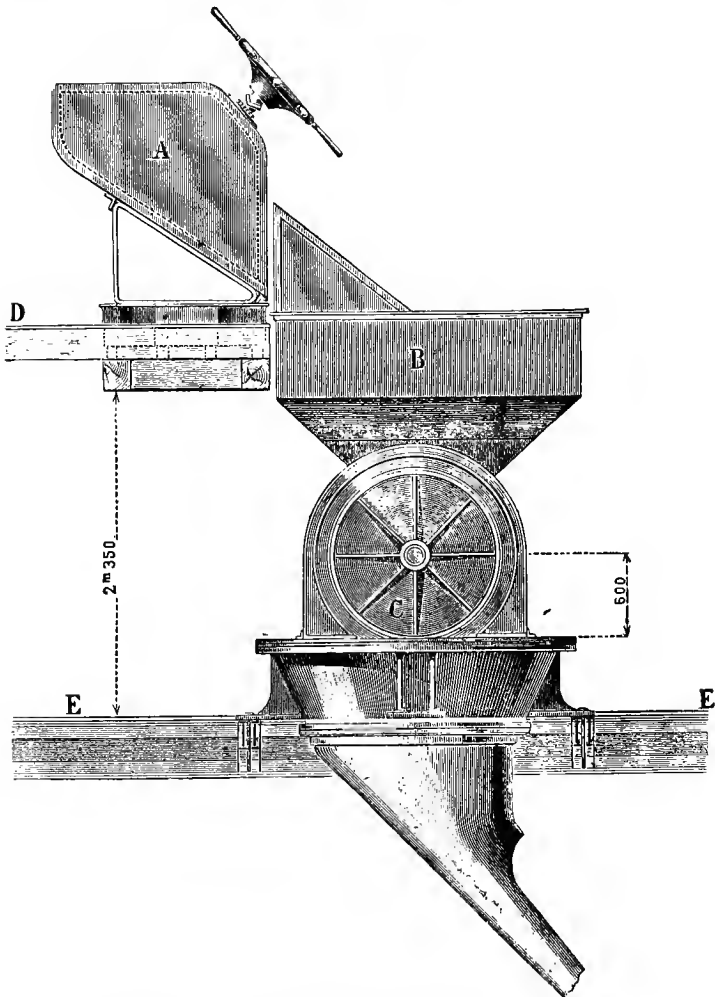


FIG. 18.—Installation of two rotary drum root-cutters on body of ordinary root-cutter. Profile.

At the extremity of the hooked piece of iron there is a cavity forming a stone eliminator, and in which pieces of beetroots, hard

substances, stones, pebbles, etc., are lodged without doing any serious damage. The stone eliminator is very simply and very rapidly emptied by a suitable arrangement.

Easily-wrought rods and a discharge door enable the beets and other bodies caught by the hook to be removed. The replacement of the knife-holders is done as rapidly as in an ordinary root-cutter. The output of this machine, in consequence of the number of knives

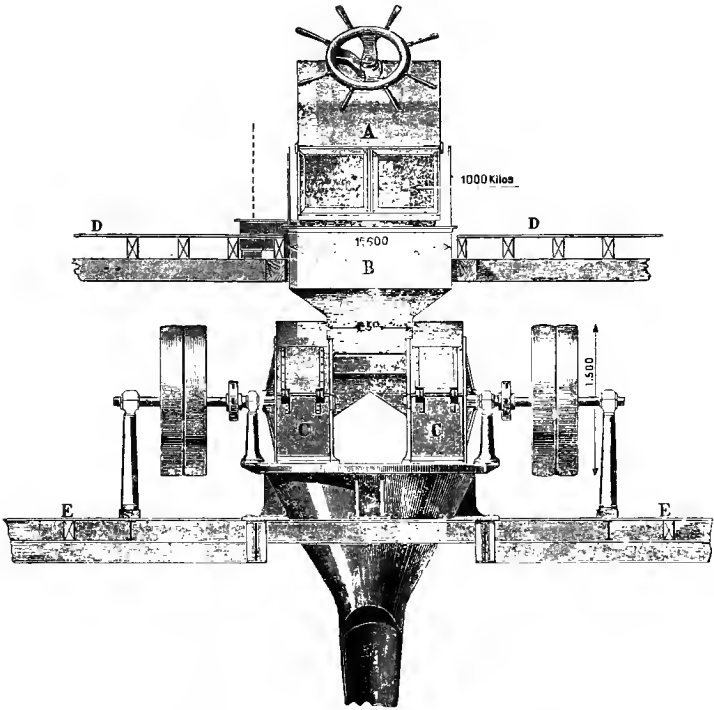


FIG. 19.—Installation of two rotary drum root-cutters on body of ordinary root-cutter.

which it contains, and the speed with which it can be driven, is very considerable. The knives, moreover, are parallel, so there is nothing to fear from the effects of centrifugal force. At 60 revolutions it easily puts through 20 tons an hour, and it is perhaps capable of revolving at 80 revolutions a minute, and even more if necessary. At 80 revolutions it does not reach the speed of the old root-cutters, and it only uses up 70 per cent. of the force necessary to work one of the latter machines. Fig. 17 shows the installation of one

of these machines mounted on the existing body of an old root-cutter, with its special hopper and weighing machine. As will be seen by the figure, it requires no cog-wheels, and leaves a large space around it for manipulating the knife-holders. Figs. 18 and 19 show the profile and the front of an installation of two drum root-cutters mounted on the existing body of an ordinary root-cutter of 2 metres in diameter. This is a very convenient arrangement, by which the production of slices may be doubled if the machines work together, and continuous working guaranteed if worked alternately. This is an advisable arrangement for factories which treat more than 400 tons of beets in the twenty-four hours.

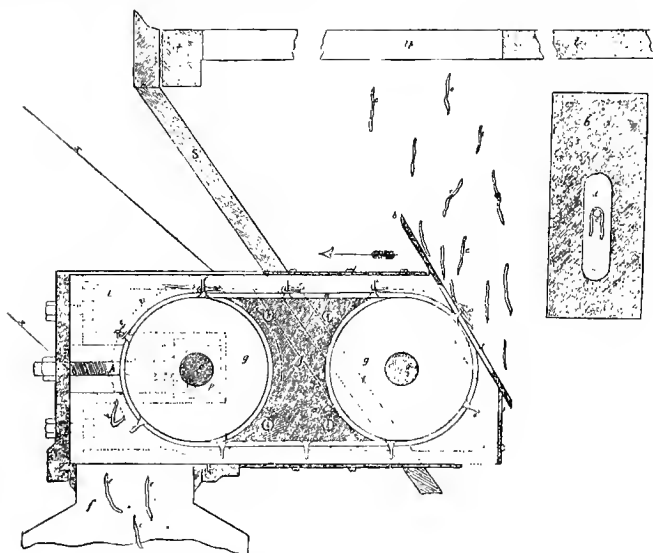


FIG. 20.—Apparatus for sampling beet slices as delivered to diffusers.

BEETROOT PULP SAMPLER

This apparatus is intended to sample automatically the beet slices as they enter into the diffusion battery. It is fixed in the chute which leads from the root-cutter to the diffuser. It consists of an inclined plane *b*, placed below the level of the root-cutter, pierced by a longitudinal hole *d*, in which a round stone *g*, carrying a strap *a* provided with spikes *C*, passes. The band *a*, passing over the stone *C*, is prolonged over a second stone *C*₁, which imparts to the belt the motion which it receives direct from some transmitted power, if the

apparatus be fixed on a root-cutter, the chute for the pulp of which is fixed, or if the fixed funnel be high enough to lodge the apparatus. Again, if the chute be used to feed a circular diffusion battery working immediately below the plane of the root-cutter, the sample may be driven by clock-work.

The distance between the stones C and C₁ being very short, the stone C₁ is fixed on a suspension platform, which always gives enough adherence to the band *a*.

The root-cutter being at work, and the band *a* also, the apparatus works as follows :—

The slices fall from the plate of the root-cutter, and some of them, falling on the inclined plane *b*, are carried on the spikes on the belt *a*. Proceeding on their journey, they fall into a reservoir *f*, which preserves them until the time they are required for analysis.

The whole of the apparatus is maintained in cast-iron plates *l*, so as to hinder other slices than those falling on the inclined plane being led into the reservoir. The longitudinal opening of the inclined plane being very narrow, it is evident that but few slices will be carried away at the same time.

The sample taken in this way enables the exact amount of sugar going into the battery to be ascertained. All that has to be done is to multiply the percentage of sugar by the weight of beetroots received at the factory. The difference between the sugar despatched from the factory plus the stock and the sugar received into the factory in the form of beets gives the loss on working.

DIFFUSION BATTERIES

Detailed description of a diffuser, size and shape.—Diffusers (Figs. 21, 23, and 24) are vertical cylinders, the capacity of which varies from 1 to 6 cubic metres. In France they are made of wrought iron of 3 to 4 cubic metres in capacity, with a height double that of their diameter.

The charging manhole on the top.—They terminate at the top in a very short cone bearing a cast-iron collarette, on which is the juice circulation tube. This collarette is closed from above by a door which is displaced horizontally round a turning pivot (Fig. 23; or upwards, Fig. 24), and is fixed in position by means of a strong screw supported by a stirrup. It is through this manhole that the diffuser is charged. It is 24 to 32 inches wide.

The shape of the bottom and its door.—The bottom of the diffusers is of very variable shape, on account of the position of the door for discharging the exhausted pulp. This door may be placed in the side (Fig. 21) or underneath (Fig. 23), or in an intermediate inclined position. When the door is on the side the bottom of the diffuser is of wrought iron, and almost flat.

The manipulation of the bottom door.—When the discharge exit is underneath, which is the best position, the door embraces the whole of the bottom of the cylinder, which then ends in a cast-iron armature which supports the door. This may be capable of being opened by horizontal displacement, as the door on the top, or from below downwards, round a hinge. It is balanced by a counterpoise (Fig. 24) in either case, so as to enable it to be handled, as it is very heavy, and it is held in position by strong bolts or screws. The handling of the bolts and screws is done by a shifting spanner, which guarantees their good working.

Inclined doors and sloping bottoms.—When the door is inclined

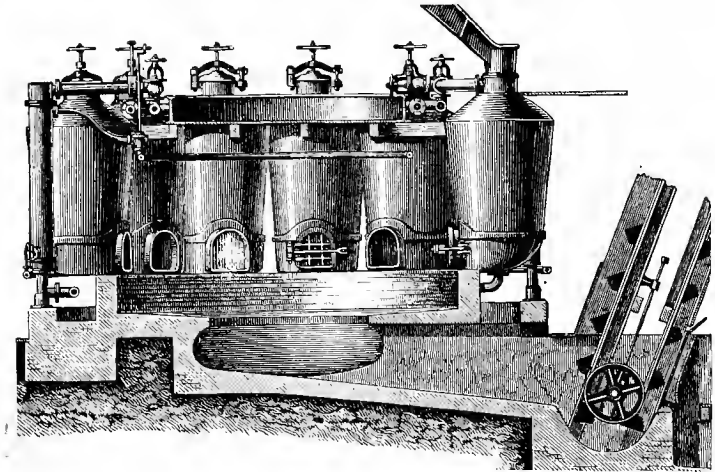


FIG. 21.—Circular diffusion battery, with inclined elevators removing exhausted pulp.

the bottom of the cylinder is all of cast iron, and the aperture itself slopes so as to facilitate the discharge of the slices.

Necessity for tight joints.—The staunchness and water-tightness of all these doors is secured by an indiarubber washer, against which the screwing-up is done. In the wide bottom doors resort has to be made to what is known as the *hydraulic joint*, i.e. the indiarubber is hollow, and, when tightening, water is injected in the interior under strong pressure, which presses the rubber very forcibly against the door.

Line batteries versus circular batteries.—The diffusers may be arranged in a straight line or in a circle.

1. *Line batteries—their general arrangement.*—This is the oldest arrangement (see Fig. 22). They are generally arranged in two parallel rows, and the discharge doors are turned in the same direction

to the inside of the double line, in such a manner that the exhausted slices fall between the two rows into a channel, in the bottom of which a helix turns and carries them away. The bottom of the channel is sometimes simply inclined with a slope sufficiently great to cause the pulp to run into a pit, from which it is extracted mechanically.

2. *Circular batteries—their many advantages.*—Circular batteries (Figs. 13, 21, 22) are very convenient, on account of the facility with which the diffusers may be filled with beet slices—a fact which will be dwelt upon later on. They are very generally adopted in France. The exhausted pulp is discharged from the diffusers into a pit, in the centre of which is an elevator or conveyor.

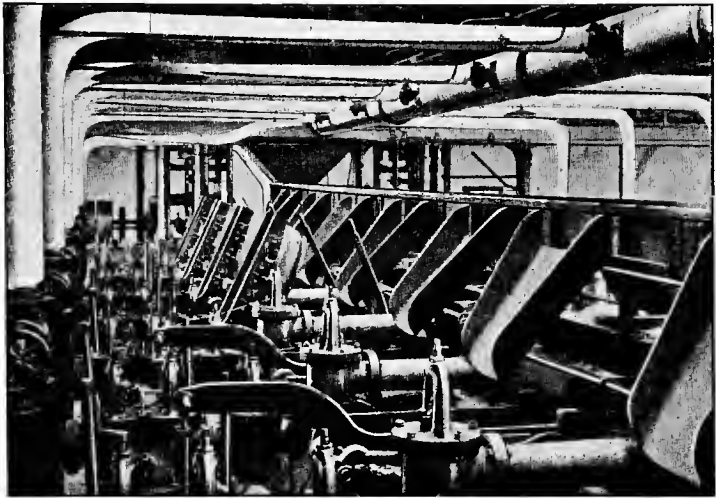


FIG. 22.—Showing the manner in which the beet slices are fed into the diffusers of line batteries (CAIL).

Charging line batteries with beet slices.—The charging of the line battery diffuser (Fig. 22) with sliced beets is done in two ways,—either (1) by means of trucks which are filled underneath the root-slicers, roll on a short aerial railway, and empty themselves from below into the diffuser; or (2) by means of a gutta-percha belt conveyor. The latter runs horizontally the whole of the length of the battery, at the bottom of a trough. The root-slicer drops the slices on the belt. In front of each diffuser the trough has a door (Fig. 22), which is opened in such a way that the slices fall into the diffuser. The door, opening inwards, makes a barricade, and prevents the slices going further on than the diffuser which is being filled.

The method of charging circular batteries.— In the circular batteries (Figs. 13, 21, and 23) quite a different arrangement is made. The root-cutter is on an upper floor. Under the root-cutter is a gigantic hopper or funnel, terminating in an inclined trough, which comes as far as the doors of, the diffusers. The funnel rests on

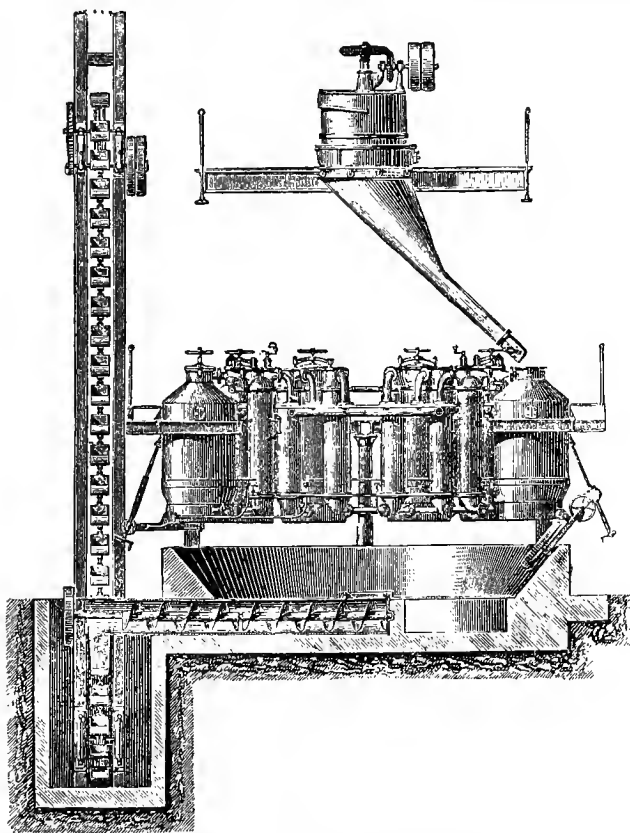


FIG. 23.—Circular battery, showing the way in which it is fed and the removal of the pulp by a helical conveyor (CALL).

bearings in such a way that it can turn on itself, and consequently the end of the trough may be brought in front of each diffuser in turn. When the root-slicer is at work the slices follow the trough right into the diffuser, without any need of any other method of transport. (Figs. 13, 17, 18, 19, and 21.)

Calorisators—their description and function.—Whether the battery be a line battery or a circular battery, each diffuser carries its own caloriser. The caloriser is a vertical bundle of pipes—surrounded by steam—in which the juice circulates as it passes from the one diffuser to the other (Figs. 23 and 72). At other times the caloriser

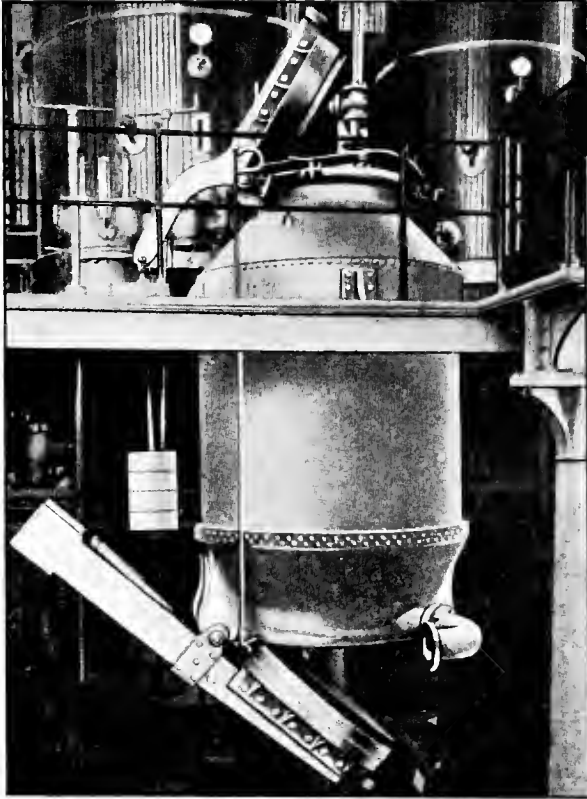


FIG. 24.—Diffuser (generally termed the "cell" of the diffusion battery, (C&L).

is simply a very wide pipe containing a large steam coil, in which the steam circulates. The caloriser is sometimes replaced, as in Belgium, by a simple injection of steam, which heats the juice in its passage through the pipe, and condenses there. This last arrangement is imposed by the Belgian Excise authorities, to avoid the leakage sometimes incidental to the other form of calorisers, and which

might injuriously affect the revenue, the sugar Excise tax in Belgium being levied on the quantity of juice produced.

The old-fashioned process of reheating the juice.—In former times, before calorisors were invented, the juice was reheated in two tanks fitted with steam coils, or in large tubular reheaters, through which the juice was passed from the one diffuser to another. These two reheaters served for the whole battery, and involved two extra taps to each diffuser. This old-fashioned method of working is still sometimes met with.

The valves all manipulated from the same stage.—All the taps of each diffuser are united in a single buffet, clearly and neatly arranged to facilitate operations. The keys of all the taps, or rather the wheels of all the valves, are thus on the same level, and at the hands of the workman whether in front or behind the diffusers (Fig. 23).

The working staff attached to a diffusion battery.—In circular batteries the battery foreman and his assistant are quite competent to do all the work required. This is also the case in line batteries with a conveyor. But, when the beet slices are conveyed to the diffusers in trucks, three or four extra men are required to work them. Notwithstanding this drawback this last system is very often preferred, because less time is lost in filling the diffusers, which is done almost instantaneously, and much more work can thus be got out of the batteries, which is a great pecuniary advantage for those factories whose diffusion equipment is on too small a scale.

ACCUMULATIONS OF AIR AND VAPOURS IN THE DIFFUSION CELLS, AND THEIR REMOVAL

This is a frequent cause of trouble. The men attending the battery should frequently blow off these accumulations. The battery man, working a battery at a high temperature, will often neglect to reduce the pressure of steam on the calorisors at the right time. The juice thus becomes heated above the boiling-point, and, when the pressure is reduced, larger volumes of steam accumulate in the cells. This steam and the air in the cells are liable to "trap," and thus prevent the sugar being extracted in a regular and uniform manner from the slices. To meet this difficulty, the appendage shown in Fig. 22 was designed by K. Leyser of Oschlersleben, Germany. It consists of a float *g*, connecting by means of a spherical joint at its upper end with the valve *b*, and guided at its lower end by means of a rod at the centre of the strainer *e*. The tube *a* communicates with the diffuser. If vapours of any kind collect in the diffuser they will pass out through the tube *a*, around the float *g*, through the valve *b* and tube *d* into the open air. Any foam that may collect in the cell will also escape. As soon as the juice in the cell rises to a

sufficient height the float *g* will lift and close the valve, and thus prevent the juice itself from escaping. The function of the small funnel is to intercept any fine pieces of pulp which may pass the

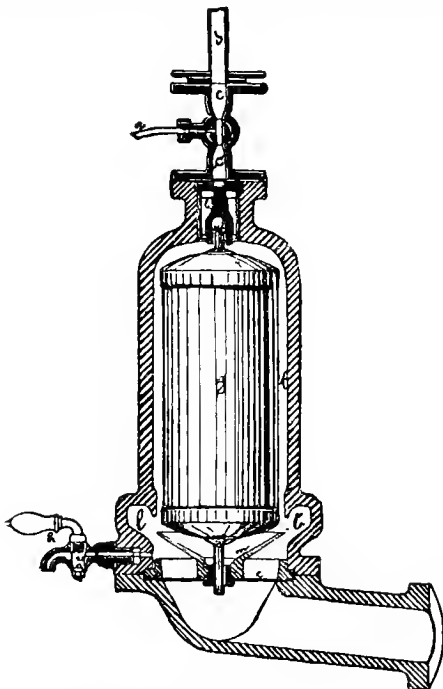


FIG. 25.—Apparatus for removing the accumulation of air and vapours in diffusion cells (K. LEYSER).

strainer and prevent them from clogging the apparatus. The funnel should be removed as occasion may require, and emptied. The valve between *cc* is closed when the apparatus is not in use.

PLAN AND ARRANGEMENT OF DIFFUSION BATTERIES

Single and double lines of batteries.—Local conditions largely control the arrangement of a battery. A single line requires a very long building, but ease of removal of its exhausted slices and favourable conditions for enlarging the plant are in favour of this system. The enormous length of the return pipes is objectionable. On the other hand, the double line facilitates the removal of exhausted slices; the return pipes are very short, and the manipulations are as simple

as in the circular battery. In both single and double line batteries there is difficulty in charging the last cell in the series with slices without either having slices left over, which fall on the floor, or giving this cell an irregular supply.

Circular batteries.—The circular battery involves a very lofty square edifice, and the methods available for removing the exhausted slices and the space occupied constitute the main drawbacks to this plan of battery.

Whatever arrangement be adopted, the actual cost of construction is very much the same in all cases.

DIFFUSION RESIDUALS—EXHAUSTED PULP

The excess of water must be removed from the water-logged pulp before it can become available as a cattle food.—Exhausted pulp, as it comes from the diffusers, is very wet owing to two reasons. The first is due to the water with which it is distended; the second is due to the physical effect on which the process of diffusion is based, in virtue of which the cells of the exhausted slices are charged with water instead of the saccharine juice which they originally contained.

Had pulp presses been in vogue, diffusion would not have been abandoned at the outset.—In this condition the pulp is of no value for cattle-feeding purposes, the moisture which it contains injuriously affecting the health of the cattle fed on it. That was the reason which led to the abandonment of Mathieu de Dombasle's process, and if pulp presses had been to the fore in those days it is more than likely that from that time forward no other system for extracting the juice would have obtained.

The many difficulties in the way of pressing the pulp.—As might be expected, the pulp is difficult to press. It has, in fact, been cooked in the diffusers at a temperature of 70° to 80° C. (158° to 176° F.) for at least half an hour; it has therefore become soft and tacky by the effect of the heat on the pectic substances exposed by the effects of slicing. All the cells under the knife being naturally opened and even often torn when the knives are blunt, is one reason why the knives should be frequently sharpened.

Its jelly-like nature a great drawback to its effectual pressure.—When a fistful of pulp is pressed in the hand it is easily converted into a jelly that strings between the fingers, more especially when the diffusion has been heated a little too much so as to work a little more rapidly. This shows the precautions which are necessary to take so as to squeeze the pulp sufficiently without spoiling it. A roller press would have no other effect than to triturate it without getting rid of the water. It is necessary, therefore, that the machine should slowly compress the pulp, and should not bruise it at the time the greatest amount of pressure is being imparted.

Kluzemann's press.—The first press which fulfilled the above requirements was Kluzemann's press. This is a vertical cylinder of cast iron resting on a cast-iron hood. In its axis there turns a piece having the form of a much elongated cone standing upright on its base of the height of the cylinder, and the base of which is slightly smaller in diameter than the cylinder. This cone carries arms almost abutting on the cylinder, in the form of wide blades and arranged in the form of a helix down to the bottom. It will be seen that, if wet exhausted pulp be placed in the cylinder, it is caught by the blades, which force it to descend and draw it into the free space between the cone and the cylinder—a space which narrows more and more on account of the conical form of the central piece. The pulp is thus slowly pressed, its water escapes through the holes in the cylinder. Moreover, when it reaches the bottom it meets a piece of cast iron which still further restricts the opening, and whose distance from the cylinder can be regulated in such a way as to still further increase the pressure.

It has to be added that the cylinder carries at intervals iron arms on the inside, which prevent the pulp from turning with the cone, and thus blocking or jamming it. The water which flows round the cylinder is collected in a gutter, and is led into a drain.

Bergneen's press.—Kluzemann's press has been altered by Bergneen (Fig. 26). The conical kernel is also made to act as a filter, and the helix is in two parts, driven at different speeds. It is a little more powerful in its effects than Kluzemann's press. Moreover, the latter has also been improved so far that there is but little difference in the amount of work got out of the two.

Selwig and Lange's press.—This press is constructed on another principle. It consists of two strong circular plates with a filtering surface fixed in a vertical frame. These plates turn slowly and in different directions with the same speed, only their axes are a little inclined, the one to the other, in such a way that they approach each other very nearly at a certain point of their circumference. If pulp be introduced between the plates at the spot where they are the furthest apart, this pulp is carried forward by the rotation of the plates, but as the pressure of the plates goes on increasing the pulp is caught and pressed, and finally escapes, sufficiently dried, after having traversed half a circumference. This press is a fine machine, extensively used. There are many other systems, but the three just described produce the best marketable pulp.

The conveyance of the pulp to the press.—The pulp is lifted out of the pit of the diffusion battery by means of cup elevators (Figs. 21 and 23). Only, the cups are perforated with numerous holes, sometimes even altogether open, so as to allow the water to escape with which the pulp is mixed in the bottom of the pit. The chain of cups discharges into a horizontal trough, at the bottom of which a

helix turns, and this trough has as many apertures as there are pulp presses. These apertures open above the hopper of each press, and may be closed when the press is not working.

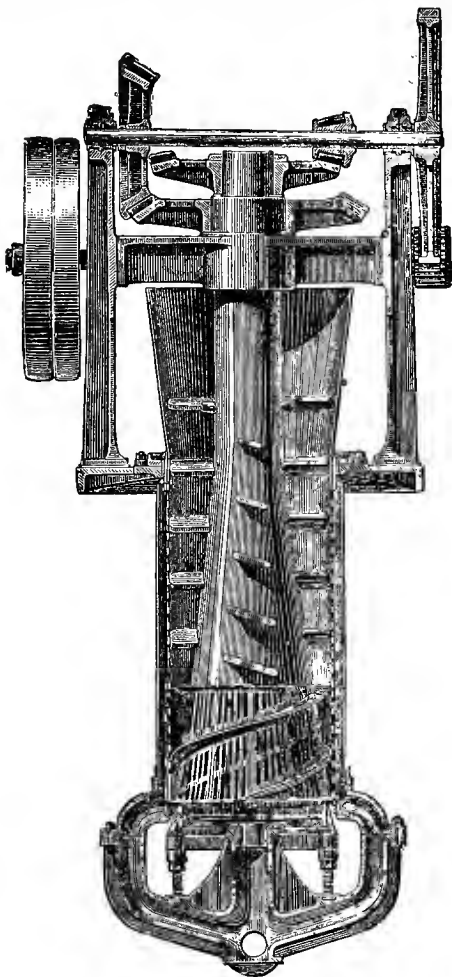


FIG. 26.—Bergneen exhausted pulp press.

The location of the pulp press floor.—The pulp presses are generally placed on a high enough level for the dried pulp to pass

into a room, below which the farmer's carts are loaded by opening a trap-door. Formerly the dried pulp was raised by a helical elevator, and discharged therefrom into the carts. In a word, the arrangements for facilitating the lifting of the pulp in different factories are endless, and dictated by circumstances and the plan of the factory buildings.

Siloing the pressed beet pulp.—When there is no immediate sale for pulp it is siloed. These silos are long deep trenches, dug out of the ground and built on the sides with potter's clay, into which the pulp is run. It is afterwards covered with earth. In the silos the small amount of sugar still left in the exhausted beet first of all enters into *alcoholic* fermentation, which afterwards gives place to the *lactic* fermentation.

The slight acidity of siloed pulp grateful to the palate of cattle.—The fermented slices very soon only consist of a homogeneous mass, their original shape quite disappears, and cattle prefer it to the fresh pulp—without doubt, on account of the slight sour taste. It is not, however, given to cattle by itself alone; it is mixed with cut straw or hay, and the animals are then very fond of it. The pulps make a valuable cattle food, says Wiley. Cattle may be fed with them in the fresh state or after preservation in silos. Lately extensive experiments have been made in drying the pulps and preserving them in the dried state, and these experiments have been fairly successful. It is stated that the value of the pulps for feeding purposes is from one-fourth to one-fifth of the value of the beets. Their feeding value may, however, be judged from the following analyses.

XXV.—ANALYSES SHOWING NUTRITION VALUE OF BEET DIFFUSION PULP (RESIDUAL).

	Fresh. Per Cent.	Pressed. Per Cent.	Siloed. Per Cent.	Pressed with Lime. Per Cent.	Dried. Per Cent.	Maceration Residues. Per Cent.
Water . . .	94.0	89.8	88.5	65.0	11.6	78.9
Ash . . .	0.4	0.6	0.9	3.9	7.1	2.8
Albumenoids . .	0.5	0.9	0.9	3.2	6.6	1.5
Cellulose . . .	1.4	2.4	2.3	9.6	19.3	4.4
Carbohydrates.	3.6	6.1	7.2	17.8	54.8	12.3
Fat . . .	0.1	0.2	0.2	0.5	0.6	0.1

The following table (XXVI.) gives the amount of spent pulp produced, in France, from 1884-85 to 1899-1900:—

XXVI.

Season.	Spent Pulp.	Price Per Metric Ton.	Season.	Spent Pulp.	Price Per Metric Ton.
1884-85 .	Metric tons. 1,207,248	fr. c. 8 61	1892-93	Metric tons. 2,192,667	fr. c. 4 96
1885-86 .	999,703	8 65	1893-94 .	2,130,669	5 16
1886-87 .	1,498,095	7 73	1894-95 .	2,907,560	4 48
1887-88 .	1,250,151	6 36	1895-96	2,192,099	4 32
1888-89 .	1,518,457	6 02	1896-97 .	2,792,548	4 35
1889-90 .	2,442,343	5 13	1897-98 .	2,704,082	4 30
1890-91 .	2,475,091	4 84	1898-99 .	2,623,277	4 19
1891-92 .	2,219,120	4 81	1899-1900	3,219,751	4 15

Ratio of weight of exhausted pulp to original weight of beets submitted to diffusion.—As the juice extracted by diffusion is replaced by an equal volume of water of unit density, the difference between the weight of the juice drawn from the diffusers and the weight of the water which replaces it will give the loss in weight which the beet slices undergo in the diffusion process. If 115 litres of juice of density 1.05—corresponding to 13.3 kilogrammes of dissolved substances per hectolitre*—be extracted on an average from 100 kilogrammes of beets, it follows that $115 \times 13.3 \div 100 = 15.295$ kilogrammes of dissolved matter have been extracted from the beet—say, in round numbers, 15 per cent. Consequently 85 per cent. of pulp remains. It will thus be seen that the quantity of exhausted pulp depends on the volume and the density of the diffusion juice, so that the coefficient to be used is not always the same, and it must be determined each time stock is taken and the factory books balanced. So, as to avoid calculation, it has generally been agreed to adopt the average figure of 85 per cent.

During manufacture the spent pulp is analysed as often as possible. It is easily sampled. The spent slices as they come from the diffuser in normal working are uniformly exhausted, and all that has to be done is to take several handfuls and mix them into a convenient sample. The samples may also be taken from the elevator cups on their way

* Kilogrammes per hectolitre=lbs. per 10 imperial gallons.
 Litres per hectolitre=imperial gallons per 100 imperial gallons.
 Grammes per bectolitre=parts by weight in 100,000 parts by volume.

to the pulp presses, taking a little from each cup. The mixed pulp is cut up, pressed, and polarised. The sugar remaining in the slices may vary, but they are frequently analysed, and the daily average fairly represents the loss of sugar in this residual.

The chemical control of the diffusion work of a beet-sugar factory during the night shift.—In most factories some little difficulty is encountered in controlling chemically the work of the night shift. The difficulty may be got over by taking advantage of the fact that there is a very close connection between the sugar left in the spent pulp and that found in the drainage from the pulp presses, so that the analyses of the latter may replace that of the spent pulp to a certain extent. To get a fair average sample of the liquor from the presses, a known quantity of basic acetate of lead is run into a litre flask, and the night foreman runs a small sample of the drainage water into the bottle from time to time in such a way that the bottle is filled by about seven o'clock in the morning. The whole is then mixed, polarised, and the dilution due to the basic acetate of lead taken into account. The easiest way is to add 100 c.c. of basic acetate solution and run in about 50 c.c. of the liquor from the presses every half-hour, so that the litre is filled by morning. The liquid is filtered after mixing, polarised, and the sugar calculated for 100 c.c. of liquid, and the result multiplied by 1.1 so as to take the dilution into account. Calculation may be dispensed with by multiplying the degrees obtained by Laurent's saccharimeter with a tube 20 centimetres long by 0.18, but it would be better to use tubes 40 or 50 centimetres in length so as to get more accurate results. In that case the coefficient will be 0.09 and 0.072 instead of 0.18.

XXVII.—ANALYSES OF WEAK LIQUORS. (SCHEIBLER'S TABLE.)

Density at 15° C. (59° F.).	Dissolved Matter.		Density at 15° C. (59° F.).	Dissolved Matter.	
	Per Cent. grammes.	Per Cent. c.c.		Per Cent. grammes.	Per Cent. c.c.
1.00000	0.00	0.0000	1.00039	0.10	0.1000
1.00004	0.01	0.0100	1.00043	0.11	0.1100
1.00008	0.02	0.0200	1.00047	0.12	0.1200
1.00012	0.03	0.0300	1.00051	0.13	0.1301
1.00016	0.04	0.0400	1.00055	0.14	0.1401
1.00020	0.05	0.0500	1.00058	0.15	0.1501
1.00023	0.06	0.0600	1.00062	0.16	0.1601
1.00027	0.07	0.0700	1.00066	0.17	0.1701
1.00031	0.08	0.0800	1.00070	0.18	0.1802
1.00035	0.09	0.0900	1.00074	0.19	0.1902

[TABLE CONTINUED.]

DIFFUSION RESIDUALS—EXHAUSTED PULP 109

XXVII.—ANALYSES OF WEAK LIQUORS—*continued.*

Density at 15° C. (59° F.).	Dissolved Matter.		Density at 15° C. (59° F.)	Dissolved Matter.	
	Per Cent. grammes.	Per Cent. e. c.		Per Cent. grammes.	Per Cent. e. c.
1·00078	0·20	0·2002	1·00234	0·60	0·6014
1·00082	0·21	0·2102	1·00237	0·61	0·6115
1·00086	0·22	0·2202	1·00241	0·62	0·6215
1·00090	0·23	0·2303	1·00245	0·63	0·6316
1·00093	0·24	0·2403	1·00249	0·64	0·6416
1·00097	0·25	0·2503	1·00253	0·65	0·6517
1·00101	0·26	0·2603	1·00257	0·66	0·6617
1·00105	0·27	0·2703	1·00261	0·67	0·6718
1·00109	0·28	0·2804	1·00265	0·68	0·6818
1·00113	0·29	0·2904	1·00269	0·69	0·6919
1·00117	0·30	0·3004	1·00273	0·70	0·7019
1·00121	0·31	0·3104	1·00276	0·71	0·7120
1·00125	0·32	0·3204	1·00280	0·72	0·7220
1·00128	0·33	0·3304	1·00284	0·73	0·7321
1·00132	0·34	0·3405	1·00288	0·74	0·7421
1·00136	0·35	0·3505	1·00292	0·75	0·7522
1·00140	0·36	0·3605	1·00296	0·76	0·7623
1·00144	0·37	0·3705	1·00300	0·77	0·7723
1·00148	0·38	0·3806	1·00304	0·78	0·7824
1·00152	0·39	0·3906	1·00308	0·79	0·7924
1·00156	0·40	0·4006	1·00312	0·80	0·8025
1·00160	0·41	0·4106	1·00316	0·81	0·8126
1·00163	0·42	0·4207	1·00319	0·82	0·8226
1·00167	0·43	0·4307	1·00323	0·83	0·8327
1·00171	0·44	0·4408	1·00327	0·84	0·8428
1·00175	0·45	0·4508	1·00331	0·85	0·8529
1·00179	0·46	0·4608	1·00335	0·86	0·8629
1·00183	0·47	0·4709	1·00339	0·87	0·8730
1·00187	0·48	0·4809	1·00343	0·88	0·8831
1·00191	0·49	0·4910	1·00347	0·89	0·8931
1·00195	0·50	0·5010	1·00351	0·90	0·9032
1·00198	0·51	0·5110	1·00355	0·91	0·9133
1·00202	0·52	0·5211	1·00359	0·92	0·9233
1·00206	0·53	0·5311	1·00362	0·93	0·9334
1·00210	0·54	0·5412	1·00366	0·94	0·9435
1·00214	0·55	0·5512	1·00370	0·95	0·9536
1·00218	0·56	0·5612	1·00374	0·96	0·9636
1·00222	0·57	0·5713	1·00378	0·97	0·9737
1·00226	0·58	0·5813	1·00382	0·98	0·9838
1·00230	0·59	0·5914	1·00386	0·99	0·9938
			1·00390	1·00	1·0039

XXVIII.—TABLE FOR THE ANALYSES OF WEAK SUGAR LIQUORS.

(French saccharimeter. Normal weight, 16.29 grammes.)

Sacchari- metrical Degrees.	Sugar per 100 c.c.		Sacchari- metrical Degrees.	Sugar per 100 c.c.	
	400-mm. tube.	500-mm. tube.		400-mm. tube.	500-mm. tube.
0.1	0.0081	0.0065	3.0	0.243	0.195
0.2	0.0163	0.0130	3.1	0.251	0.201
0.3	0.0243	0.0195	3.2	0.259	0.208
0.4	0.0326	0.0260	3.3	0.267	0.214
0.5	0.0405	0.0325	3.4	0.275	0.221
0.6	0.0486	0.0390	3.5	0.283	0.227
0.7	0.0567	0.0455	3.6	0.291	0.234
0.8	0.0648	0.0520	3.7	0.300	0.240
0.9	0.0729	0.0585	3.8	0.308	0.246
			3.9	0.316	0.253
1.0	0.081	0.065	4.0	0.326	0.260
1.1	0.089	0.071	4.1	0.332	0.266
1.2	0.097	0.074	4.2	0.340	0.273
1.3	0.105	0.084	4.3	0.348	0.279
1.4	0.113	0.091	4.4	0.356	0.285
1.5	0.121	0.098	4.5	0.364	0.292
1.6	0.129	0.104	4.6	0.373	0.299
1.7	0.138	0.110	4.7	0.381	0.305
1.8	0.146	0.117	4.8	0.389	0.312
1.9	0.154	0.124	4.9	0.397	0.318
			5.0	0.405	0.325
2.0	0.163	0.130	5.1	0.413	0.331
2.1	0.170	0.137	5.2	0.421	0.338
2.2	0.178	0.143	5.3	0.429	0.344
2.3	0.186	0.150	5.4	0.437	0.351
2.4	0.194	0.156	5.5	0.445	0.357
2.5	0.202	0.162	5.6	0.453	0.364
2.6	0.210	0.169	5.7	0.461	0.371
2.7	0.218	0.175	5.8	0.469	0.377
2.8	0.227	0.182	5.9	0.477	0.383
2.9	0.235	0.188	6.0	0.486	0.390

The analysis of the spent beet slices is somewhat complicated. The spent slice is a very watery body of a gluey-like nature, from which it is difficult to press all the liquid which it contains out of it. A different method of analysis has to be adopted to that used in the case of the beet itself. By means of a mince-meat knife the slice is divided into as small fragments as minutely as practicable, and a known weight of the substance is weighed out into a basin, water

added, and heat applied. The sugar is diffused into the water. The liquid is run off into a graduated flask, the pulp is again washed with boiling water, which is likewise run into the flask, by which time the pulp is sufficiently exhausted. In any case, as the pulp does not contain much sugar, the error is trifling. The liquid is defecated, made up to the mark, filtered, and polarised in a 400 or 500 millimetre tube. A rule-of-three sum gives the percentage of sugar in the pulp.

It is more advisable, as in Pellet's method (p. 59), to introduce the pulp itself into the flask, heat with water, and deduct the volume due to the woody fibre. The loss of sugar may be calculated from the weight of the spent pulp and that of the wash-water, if they be known, and their analysis. Such calculations are, of course, mere estimations; moreover, it is a matter of little or no consequence. If the analysis gives the lowest possible results, that is all that is necessary. All that has to be done is to determine whether the spent pulp contains more or less sugar, so that the battery foreman may work his battery in such a way as to prevent as little loss as possible, or at least limit himself to a figure agreed upon between himself and the works manager, who should find out whether the cost of extracting the sugar remaining in the pulp does not cost more than the value of the sugar itself. A good manager endeavours to extract as much sugar from the beet as possible, but he should not go too far, as the cost of production then exceeds the selling price. It all depends on the attention paid to these matters whether the manager is a good or bad administrator of his factory.

XXIX.—EXHAUSTED PULP.

(Juice of exhausted pulp. 400-millimetre tube. French saccharimeter. Normal weight of instrument, 16·2 grammes; addition of $\frac{1}{10}$ basic acetate of lead.)

Sacchari- metrical Degrees.	Sugar. Per Cent.	Sacchari- metrical Degrees.	Sugar. Per Cent.	Tenths.	
				Degrees.	Sugar. Per Cent.
1	0·09	11	0·98	0·1	0·01
2	0·18	12	1·07	0·2	0·02
3	0·27	13	1·16	0·3	0·03
4	0·36	14	1·25	0·4	0·04
5	0·45	15	1·34	0·5	0·05
6	0·53			0·6	0·06
7	0·62			0·7	0·07
8	0·71			0·8	0·08
9	0·80			0·9	0·09
10	0·89				

XXX.—EXHAUSTED PULP.

(Table for German instruments. Juice of exhausted pulp. 400-millimetre tube. Normal weight of instrument, 26·048 grammes ; addition of $\frac{1}{10}$ basic acetate of lead.)

Sacchari- metrical Degrees.	Sugar. Per Cent.	Sacchari- metrical Degrees.	Sugar. Per Cent.	Tenths.	
				Degrees.	Sugar. Per Cent.
1	0·14	10	1·43	0·1	0·02
2	0·29	11	1·57	0·2	0·03
3	0·43	12	1·72	0·3	0·04
4	0·57	13	1·86	0·4	0·05
5	0·72	14	2·00	0·5	0·07
6	0·86	15	2·14	0·6	0·08
7	1·00			0·7	0·10
8	1·14			0·8	0·11
9	1·29			0·9	0·15

PFEIFFER'S AUTOMATIC DISCHARGE DIFFUSION PROCESS

By the adoption of this process the following plant items may be dispensed with, namely, the lower doors of the diffusers, the pulp helix, the pit, and the pulp elevator and the wash-liquor pump.

In the present diffusers, with bottom discharge (Fig. 24), the lower door of the diffusers is about equal to the diameter of the body of the diffuser. It is advisable, therefore, to fix a rather narrow limit, so as not to exceed a maximum height of beet slices, whatever may be the capacity of the diffuser, so as to obtain on drawing off from underneath a liquor of high density, easy and uniform circulation, and the most satisfactory exhaustion. The central entrance and exit of the juice facilitate these results, but it is evident that the juice, entering by the centre of the upper door, passes preferably through the beet slice nearest its entrance, and that at the expense of the slices further away alongside of the periphery. Analogous phenomena occur at the exit of the juice, from the diffuser ; all the circulation channels converge towards the exit orifice, in the centre, in such a way that the best exhausted portion in the mass of the diffuser assumes a form resembling that of an egg. That should therefore be the form of the theoretical diffuser. This natural circulation is avoided by increasing the surface perforation of the centre towards the periphery, so as to ensure a uniform entrance of the juice on the plane of the beet slices, limited by the perforated wrought-iron plates, but the calculation of the best perforations to adopt is based on approximations without exact data.

This preamble is necessary to bring out the advantages of the conical form imparted to the fixed bottom of the diffusers, adopted for automatic discharge in Pfeiffer's patent (Fig. 27). This bottom, the

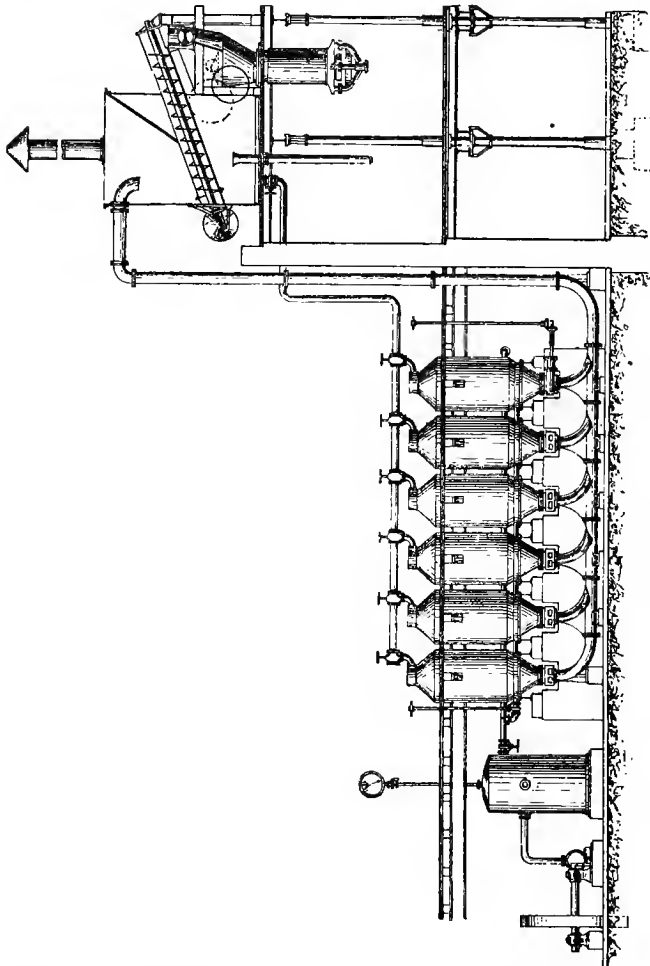


FIG. 27. — Pfeiffer's automatic discharge diffusion process (CALL).

slope of which has been determined on as the practical result of two years' trials and two years' actual working, is lined with perforated wrought iron ; a discharge orifice placed at the lower part of the cone,

and consequently in the centre, induces the circulation as in the ordinary diffuser. On this double bottom is a crown, closed by a valve, manipulated from the upper floor, through which air enters during the discharge. A tight valve, with a hydraulic joint, likewise manipulated from above, closes the beak of the cone, which connects further on with the cast-iron piping passing through the axis, and below the diffusers, and rising again directly to open in the discharge tank placed above the pulp presses.

This tank is divided into two separate superimposed parts: the upper part retains the pulp, which is distributed regularly to the presses; the double bottom collects the wash-waters, the overflow of which runs on to the washers, and the portion kept in reserve below the level, regulated by the overflow, is used before each discharge to fill the discharge pipe, partially emptied by the preceding operation.

A compressor, the capacity and the force of pressure of which are determined according to the size of the battery and the height of the presses, forces into a recipient a suitable volume. A manometer placed on the recipient, and brought well within sight of the battery, indicates the pressure of the air—say, to fix ideas, 2 kilogrammes; as soon as this pressure is reached, an arrangement, acting on the distribution of the compressor, enables the latter to discharge its functions without absorption of work.

In discharging, the diffuser being isolated, the lower valve is opened, and also the wash-liquor valve, to fill the pipe. The latter is almost at once closed, and the compressed air is turned on. In all, three valves are manipulated from the upper floor; the operator watches his manometer, the pressure of which rapidly descends to 0, which then indicates that air is passing through the pipe into the discharge tank. The operation, which lasts 30 seconds in the case of a diffuser of 50 hectolitres (1100 gallons), is finished, the compressed air is turned off, the discharge valve closed, and the upper door is opened for the refilling of the diffuser with fresh beet slices.

The operation, as will be seen, is very simple, and the battery may thus be laid down on very slight masonry, the pit being suppressed. The greatest cleanliness is ensured below the stage of the diffusers, where no trace of water, juice, or pulp is to be seen; the operation goes on regularly and without breakdowns.

PELLHARDT'S ELIMINATOR OF PULP FROM DIFFUSION JUICE

This machine for eliminating the pulp from the diffusion juice retains the stray pulp and the pieces of pulp carried with the juice into the measuring vessels. It therefore frees the juice from all solid substances, which it holds in suspension, and which increase the volume and weight shown by the hydrometer. It increases the purity of the juice, facilitates the working of the carbonatation process, and

does away with the annoyances engendered by the conveyance of the pulp into the reheaters, carbonators, etc., where it forms deposits. It is claimed that this is the only machine which is really practical. It works without interrupting the circulation of the juice in the diffusion battery, and does not require any cleaning. That is the reason why it is the only one the use of which is authorised by the Belgian Excise, where the tax is levied on the diffusion juice.

Description.—It consists (Fig. 28) of a cylinder ending in a small cone, of a peculiarly-shaped perforated mobile cylinder, forming a scraper and a butterfly valve for discharging, and it is driven by cog-wheels and pulleys. The juice from the battery runs into the machine through a pipe half-way up the sieve, and passes into the interior of the perforated cylinder. From there it flows through an elbow into

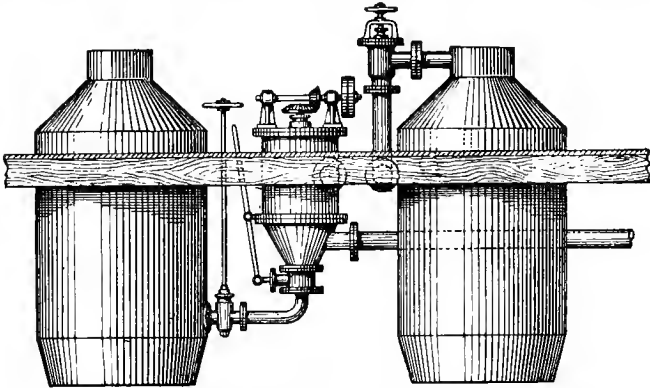


FIG. 28.—Showing Pellhardt's pulp eliminator fixed between two diffusers (MAGUIN).

the pipe leading into the measuring vessels. The pulp drawn over with the juice is retained by the revolving perforated cylinder and accumulates in the conical part, in which it is discharged by the manipulation of a valve in one of the diffusers of the battery. Care is taken not to empty the machine except when the diffuser in question is half filled with fresh beet slices. This perforated cylinder revolves at a speed of 10 to 12 revolutions a minute. A special scraping arrangement continually removes the pulp from the cylinder.

Installation.—The machine may be fixed on the stage of the diffusion battery between the juice pipe from the battery to the measuring tanks. The discharge pipe should be at a third of the height of the diffuser with which it is connected. This pipe should have an internal diameter of 70 to 80 millimetres ($2\frac{3}{4}$ to 3 inches), and be closed by a valve or tap fixed to the body of the diffuser. The butterfly valve is wrought by a rod worked from the

diffusion stage. The working of the machine requires little supervision. It is not opened to remove the deposit. There is therefore no interruption in the circulation. It is discharged by opening the tap fixed to the diffuser at the moment when the latter is almost half filled with fresh beet slices. Then the butterfly valve is opened and the deposit flows into the diffuser. When the machine is empty, the valve is first closed and then the tap. The inside scraper should touch the perforated cylinder. With this end in view, it is drawn near to it by two outside screws fixed in the outside cylinder of the machine. Care is taken to open the small air-tap fixed to the lid, so as to let the air escape.

CHAPTER V

DEFECATION AND CARBONATATION—PURIFICATION OF THE JUICE

THE PROPERTIES AND NATURE OF THE JUICE

CRYSTALLISATION or concentration prevented by gelatinisation of pectic substances.—The juice of the sugar beet is a more or less deep violet-coloured turbid liquid, with a fresh smell when extracted from sound beets. The crude unrefined juice cannot be evaporated, because it contains a large number of pectic, albumenoid, and other impurities, which transform it when heated into a kind of gelatinous confection or jelly, from which it would be well-nigh impossible to extract the smallest amount of sugar. The term jelly is used advisedly, because it is owing to the pectic matters of fruits that these sweetmeats assume the gelatinous form.

Defecation.—*Elimination of the pectic substances from the juice by lime.*—The manufacture of sugar from the juice of the sugar cane being a much older process than the extraction of sugar from the juice of the sugar beet, it was quite natural that the process used in the purification of the beet juice should have been copied from the more ancient sister industry. This purification of the cane juice was effected in the sugar-cane-growing countries by simply boiling the juice with lime. The same process was extended to beet juice. The process is termed *defecation*.

The action of lime on the juice of the sugar beet.—When a small quantity of lime, say, a few thousandth parts, is incorporated with beet juice, the lime is observed to coagulate certain principles of the juice, and this coagulum is eventually precipitated to the bottom of the vessel, whilst the supernatant juice is converted into a straw-yellow and perfectly limpid liquid. This process is, however, a tedious and prolonged one, requiring a certain amount of time before it operates effectually.

However, by boiling the juice, treated with lime in the manner indicated, a thick layer of scum is formed which floats on the surface, whilst at the same time a rather heavy coagulum is precipitated to the bottom of the vessel, and the intervening liquid becomes perfectly limpid in a few minutes. The defecation of the juice is accomplished. In actual practice, jacketed pans were used, into which the

juice and lime were run, and the whole brought to the boil, the scum skimmed off, and then, when precipitation was complete, the clear limpid liquid was run off.

The liquid from the jacketed defecation pans was evaporated, then boiled over a naked fire until it assumed a syrupy condition, when it was run into crystallising basins or moulds, where it was left to drain, and finally clarified or refined, to free it from molasses.

Defecation was a long process. Many attempts were made by chemists to improve the method of conducting it, the more so as it was so liable to failure and mishaps.

1792. *Achard's defecation process*.—So far back as 1792, Achard attempted to defecate sugar-beet juice by sulphuric acid instead of lime. The process gave good results, but it was dangerous, as a small trace too much of acid completely destroyed the sugar.

1825. Later on, *Mathieu de Dombasle*, then *Chaptal*, and lastly *Dubrunfaut*, about the year 1825, used sulphuric acid in conjunction with lime, so as to saturate or neutralise the excess of lime without rendering the juice acid.

1849. *Rousseau's carbonatation process*.—The above process was very successful, and lasted until about 1849, when it was superseded by Rousseau's process.

Description.—Rousseau's process consisted in following the lime-treated juice, after decantation, by treating it with carbonic acid gas, from a charcoal furnace, until the excess of lime was saturated. This idea was first suggested by Kuhlmann in 1833, again taken up by Barruel, and finally brought to a successful issue by Rousseau. Rousseau's process was rapidly adopted in sugar factories. It was more economical than Dubrunfaut's, because it effected a great saving in bone char, the carbonate of lime being much less soluble in sugar solutions than the sulphate (see Table).

XXXI.—SOLUBILITY IN SUGAR SOLUTIONS OF CERTAIN INSOLUBLE SUBSTANCES (JACOBSTHAL).

Solubility in a Sugar Solution of—	5 Per Cent.	10 Per Cent.	15 Per Cent.	20 Per Cent.	25 Per Cent.
Calcium sulphate . . .	2 095	1 946	1 593	1 539	1 333
„ carbonate . . .	0 027	0 036	0 024	0 022	0 008
„ oxalate . . .	0 033	0 047	0 012	0 088	0 001
„ phosphate . . .	0 029	0 028	0 014	0 018	0 005
„ citrate . . .	1 813	1 578	1 505	1 454	1 454
Magnesium carbonate . . .	0 317	0 199	0 194	0 213	0 284

1859. *Perier and Possoz's double carbonatation process*.—*Jelinek's process*.—Perier and Possoz, in 1859, in conjunction with the Cail

Engineering Company, brought out the double carbonatation process, now adopted by all beet-sugar factories throughout the world.

In Austria this system, almost without modification, is known as the Jelinek process; it is also known as the turbid defecation process; but the process, however known, involves the same method of working, which has, nevertheless, been slightly varied from time to time.

The chemical composition of beet juice.—In order to understand the chemical reactions and phenomena involved in the process of defecation, it is necessary first of all to understand the composition of the sugar beet.

The detailed chemical composition of the beet and the beet juice is given in the accompanying table.

XXXII.—COMPOSITION OF BEETROOT.

Water, 79 to 84·5 per cent.

Dry matter, 15·5 to 21·0 per cent., of which— } Juice.

Soluble in water, 11·5 to 17·0 per cent.

Insoluble in water, 4 to 5 per cent.

Ingredients of juice—

I. Water 80 per cent.

II. Dry matter 20 ,,

A. Sugar 15 per cent.

B. Non-saccharine matter 5 ,,

(a) Ash, 0·8.

(1) Incombustible salts—

Potassium, sodium, rubidium, vanadium, calcium, magnesium, iron and manganese, combined with chlorine, sulphuric, phosphoric, silicic, and nitric acids.

(2) Salts transformed by combustion into carbonates.

The same metals combined with oxalic, citric, malic, and succinic acids.

(b) Nitrogenous matter, 1·60 per cent.

Proteins (albumen, etc.).

Plasmatic substances.

Asparagin ($C_4H_8N_2O_3$), and various amides.

Betaine ($C_5H_{11}N_2O_2 + H_2O$).

Glutamine ($C_3H_5(NH_2)(CONH_2)CO_2H$).

Leucine ($NH_2C_6H_{10}CO_2H$).

Tyrosine ($C_6H_4(OH)(C_2H_5(NH_2)CO_2H$)).

(c) Non-nitrogenous bodies, 1·0.

Raffinose ($C_{18}H_{32}O_{16} + 5 H_2O$).

Arabinose ($C_6H_{10}O_5$)¹².

Dextrine ($C_6H_{10}O_5$)¹².

Soluble pectic substances.

Chlorophyll.

Chromogene.

Fat.

Coniferin ($C_{16}H_{22}O_8 + 2 H_2O$).

(Vanillin, $C_8H_8O_3$).

(d) Cellulose bodies, 1 per cent.; pectic, 0·6 per cent., and colouring substances.

(e) Cholesterin ($C_{26}H_{43}OH + Aq$).

The analysis of weighed versus measured quantities.—Although the processes used in the analyses of beet juice are uniform in all countries, yet the method of expressing or reporting the results are not identical. In Germany all results are brought or calculated to 100 parts by weight, *i.e.* grammes per 100 grammes of juice. In France, on the other hand, the analytical results are expressed in kilogrammes per 100 litres of juice, *i.e.* per 100 parts by measure (volume). The German results are simply the French results divided by the density. In most instances it is more rational to bring the figures to the volume of the juice, as it is the only factor known, and is deduced from the capacity of the containing vessels. But, on the other hand, where temperature comes into play, the percentage by weight is the most rational course to follow in expressing the results. It is therefore advisable, in the analyses of diffusion juice, to determine the weight of the juice per 100 kilogrammes of beet; the weight does not vary with the temperature. In beet analysis by the indirect method the sugar per cent. of juice ought always to be brought to 100 parts by weight of grammes of juice, and not to 100 c.c., the volume of juice having no constant ratio with the weight of the beets.

Determining the density of the juice.—The use of hydrometers.—The density may be taken by Mohr's hydrostatic balance, an accurately graduated, as well as sensitive, hydrometer, or any other suitable instrument, as the special areometers of Brix or Balling graduated into tenths of a degree.

Influence of temperature.—It is necessary to work at the temperature at which the instrument is graduated—a condition often difficult to realise; use is therefore made of correction tables (see Table XVII. p. 50). The degrees Brix show the apparent percentage of dissolved substances.

The actual percentage of sugar is determined by direct polarisation if the juice be sufficiently bright, otherwise it is treated with a 10 per cent. solution of basic acetate of lead and filtered. The results are either multiplied by 1.1, or the observation is made in a 220-millimetre tube.

The polarimetric degrees are multiplied by 1.629 for the French saccharimeter, and 2.6048 for the German saccharimeter, and the result then expressed in grammes (*a*) per 100 c.c., or (*b*) per 100 grammes of juice.

All calculations may be dispensed with by using a flask graduated in three different places, namely 100, 110, and 128 c.c. 100 c.c. of juice are drawn off, basic acetate of lead added to about 110, or less, as occasion may require, and then brought up to the mark of 122.8 c.c.

The filtrate contains exactly the normal French weight, 16.29 grammes in 100 c.c., so that by dividing by 5 or multiplying by 2 the percentage of sugar is obtained.

When a sugar beet is pulped and the juice pressed, it will be seen from the table, that besides the sugar and water the juice contains soluble mineral salts, soluble albumen, and other organic bodies, such as certain acids, asparagin, etc.

The pectic and pectose fermentation of the unpurified juice.—If the crude juice as it comes from the press be allowed to stand for some time, the pectic matters enter into a species of fermentation, which transforms it into two gelatinous acids—the pectic and the pectosic. The juice then aggregates into a jelly, or, if it be diluted with water, it strings, like certain white wines, after they have gone wrong.

Secondary fermentations.—For that reason, therefore, the juice cannot be kept for any length of time, the more so as secondary or complementary fermentations set in, which change the sugar into uncrystallisable glucose or mannite.

Beneficial effect of liming the juice.—If lime be incorporated with the cold juice the organic acids are saturated by the lime, and the pectic bodies are changed into pectate of lime, which is precipitated to the bottom of the vessel. The liquid thus treated may, so to speak, be preserved indefinitely, provided the lime be not in excess. In fact, Maumené suggested this method for storing the juice in tanks, and extracting the sugar from it all the year round.

Bad effect of an excessive application of lime.—*Production of lactic and butyric acid fermentation.*—But if lime be used in very great excess, a special fermentation process, due to the albumenoids, namely, the lactic fermentation, is developed, followed by the butyric fermentation. The latter gives off a most disgusting nauseous smell. This is the cause of the bad smell of the recesses of badly kept sugar factories.

The effect of boiling the juice with and without lime.—When the crude juice is boiled without lime the albumen is coagulated; the pectic matters are unaffected if the ebullition be not carried too far. But when the juice is boiled in presence of lime both the albumenous and the pectic impurities are precipitated, as well as certain organic lime salts. This constitutes the *Defecation Process*, properly so-called.

Defecation.—*The rationale of the process.*—Should the lime not be used in too great excess, the dose being calculated so as to precipitate the organic impurities alone, a clear limpid juice is obtained by this method. On the other hand, the juice is turbid if the lime be used in excess. The cause of the turbidity of hot juices, when treated with an excess of lime, is due to the action of the lime on the sugar. The sugar, in fact, combines with the lime to form *sucrate* of lime. Now there are several compounds of sugar with lime, according to the number of equivalents of lime combined with one equivalent

of sugar. *Monobasic*, *dibasic*, and *tribasic* sucrates of lime are known. *Monobasic* salts containing one equivalent of base are known as *normal* salts, whilst those with more than one equivalent of base are termed *basic* salts—*e.g.* basic acetate of lead.

Solubility of the monobasic, dibasic, and tribasic sucrates of lime.—Both the *monobasic* and the *dibasic* sucrates of lime are soluble in water, but the *tribasic* sucrate of lime is insoluble. When a solution of a soluble sucrate is boiled it becomes turbid, owing to the formation of tribasic sucrate. From numerous experiments made by Horsin-Deon it would appear that the effect of heat is to cause the lime to combine with the least possible amount of sugar, and, as under these circumstances the sucrate containing the least amount of sugar is the tribasic sucrate, it is this sucrate which is formed and precipitated.

The formation of the tribasic sucrate to be avoided.—Consequently, if a juice limed in this way be filtered whilst it is kept in ebullition, a portion of the sugar will remain on the filter in the form of sucrate. The formation of the tribasic sucrate is therefore a danger that has to be guarded against, so as not to lose sugar in the sludge. A very important point to observe, however, is that the tribasic sucrate redissolves in the excess of sugar when the juice cools.

All sucrates of lime unstable compounds.—Sucrate of lime, in whatever state of combination it exists, is a very unstable combination. The least acidity decomposes the combination in all cases; sulphuric acid, carbonic acid, and alkaline carbonates easily decompose it, with formation of the corresponding lime salt.

The practical application of the decomposition of sucrates of lime by CO₂.—By injecting carbonic acid gas, therefore, into a juice rendered turbid by sucrate of lime, insoluble carbonate of lime is precipitated and sugar is liberated. This is the inherent principle involved in this reaction, which has made Rousseau's process such a success, because there is no longer any need to fear an excess of lime in the juice, carbonic acid being ready on the spot to cope with and neutralise it so as to prevent any possible loss of sugar from this excess.

The sucrocarbonates and the risk involved in their formation.—But there still remains an unforeseen element of danger in Rousseau's process. This is the *solubility of carbonate of lime in sugar*, and the formation of an insoluble sucrocarbonate which, passing into the sludge, involves a loss of sugar.

Boivin's and Loiseau's researches on the sucrocarbonates.—Boivin and Loiseau, about the year 1868, made a very interesting examination of a compound of sugar, lime, and carbonic acid, which they termed the hydro-sucrocarbonate of lime ($6 \text{ CaO}, \text{C}_{12}\text{H}_{22}\text{O}_{11}, 3 \text{ CO}_2$), in which *one* equivalent of sugar was found in combination with *six*

equivalents of lime and *three* of carbonic acid. This substance forms a jelly in the midst of sucro-calcareous solutions, which, if they are limpid, become turbid after having absorbed a certain amount of carbonic acid gas. Moreover, an excess of carbonic acid gas decomposes them. These facts had been observed, without any attempt at explanation, as far back as 1825, by *Dubrunfaut*. The subject was again taken up later on by *Feltz*, simultaneously with *Boivin* and *Loiseau*, whilst in 1871 *Horsin-Deon* made an exhaustive examination of the phenomenon which threw complete light on the whole question.

The theory of the formation of sucrocarbonates.—The combined result of all these researches was to demonstrate that, as soon as a certain amount of carbonic acid gas has been injected into a limed juice, a certain amount of hydro-sucrocarbonate of lime is precipitated, and that it is indispensable to continue the carbonation to destroy it, and that, if the operation be not pushed far enough, a certain portion of sugar is lost in the sludge. It is therefore necessary to ascertain the right point of saturation, so as to avoid all loss of sugar in the form of sucrocarbonates. This point has been very well determined since then by the help of the laboratory. But, at the time of *Rousseau's* process, chemistry found but little favour or encouragement in sugar factories, so that, working without a guide, the manufacturer was exposed to mishaps which he was powerless to prevent.

Summary.—The defecation of the juice is therefore effected by those reactions which occur when it is treated with lime and carbonic acid. The sucrate of lime which is formed liberates the sugar from the organic substances, which are thus free to combine more easily with the lime in excess, the sugar having now lost its solvent action on them. Moreover, the carbonate of lime which is formed acts like the base of a lake on certain soluble impurities—amongst others the colouring principles, which are thus incorporated with the lime, hence the dark colour of the sludge. Finally, the sucrocarbonate which was formed during these lateral reactions is destroyed in its turn, and the last reactions of the nascent carbonate of lime completes the purification of the juice.

Failure of all attempts to supersede lime in the purification of the juice.—The several combinations which sugar forms with lime, and which are afterwards decomposed so as to liberate the sugar when the purification of the sugar is complete, are highly interesting, and to them are due the success of the use of lime in the purification of the juice. Notwithstanding the many attempts to find a substance or substances which might advantageously replace lime and carbonic acid, these two reagents still remain the only purifying agents in general use in sugar factories.

XXXIII.—TABLE GIVING THE QUANTITY OF MILK OF LIME TO ADD PER HECTOLITRE OF JUICE EXTRACTED (UNGER).

(By means of this table the quantity of quicklime to be added in the form of milk of lime may easily be ascertained.)

Quicklime per cent. to be added.	Degrees Baumé.										
	10	11	12	13	14	15	16	17	18	19	20
	Litres.										
2.00	21.2766	19.2307	17.3913	15.8730	14.5985	13.5135	12.5786	11.7647	11.0497	10.3627	9.7087
2.10	22.3404	20.1923	18.2609	16.6667	15.3285	14.1892	13.2075	12.3529	11.6022	10.8808	10.1942
2.20	23.4043	21.1538	19.1305	17.4603	16.0584	14.8649	13.8365	12.9412	12.1547	11.3990	10.6796
2.30	24.4681	22.1154	20.0000	18.2540	16.7883	15.5405	14.4654	13.6294	12.7072	11.9171	11.1650
2.40	25.5319	23.0769	20.8696	19.0476	17.5182	16.2162	15.0943	14.1176	13.2597	12.4352	11.6505
2.50	26.5957	24.0385	21.7392	19.8413	18.2482	16.8918	15.7232	14.7058	13.8122	12.9533	12.1359
2.60	27.6595	25.0000	22.6088	20.6349	18.9781	17.5674	16.3521	15.2941	14.3046	13.4710	12.6213
2.70	28.7233	25.9611	23.4784	21.4286	19.7080	18.2430	16.9810	15.8823	14.9171	13.9895	13.1068
2.80	29.7871	26.9231	24.3480	22.2222	20.4379	18.9189	17.6100	16.4706	15.4696	14.5077	13.5922
2.90	30.8509	27.8847	25.2174	23.0159	21.1678	19.5945	18.2389	17.0588	16.0221	15.0259	14.0777
3.00	31.9149	28.8463	26.0870	23.8095	21.8978	20.2701	18.8678	17.6471	16.5746	15.5439	14.5631
3.10	32.9787	29.8078	26.9566	24.6032	22.6277	20.9458	19.4967	18.2353	17.1271	16.0620	15.0485
3.20	34.0426	30.7694	27.8262	25.3968	23.3576	21.6214	20.1256	18.8235	17.6796	16.5802	15.5340
3.30	35.1064	31.7308	28.6958	26.1904	24.0876	22.2970	20.7547	19.4117	18.2321	17.0984	16.0194
3.40	36.1702	32.6924	29.5652	26.9841	24.8175	22.9726	21.3836	20.0000	18.7845	17.6165	16.5048
3.50	37.2340	33.6539	30.4348	27.7777	25.5174	23.6483	22.0125	20.5882	19.3370	18.1346	16.9803
3.60	38.2979	34.6154	31.3044	28.5714	26.2774	24.3243	22.6415	21.1764	19.8895	18.6527	17.4757
3.70	39.3617	35.5770	32.1740	29.3650	27.0073	24.9999	23.2704	21.7647	20.4420	19.1709	17.9611
3.80	40.4255	36.5386	33.0436	30.1587	27.7372	25.6755	23.8993	22.3529	20.9944	19.6891	18.4465
3.90	41.4894	37.5000	33.9132	30.9523	28.4671	26.3511	24.5282	22.9412	21.5469	20.2073	18.9320
4.00	42.5532	38.4616	34.7828	31.7460	29.1971	27.0268	25.1572	23.5294	22.0994	20.7254	19.4175

	21	22	23	24	25	26	27	28	29	30	Example.—For a juice extraction of 116.9% a lime addition of 3% the quantity of milk of lime to be added per hectolitre of juice is 14.4631 = 12.458 litres of milk of lime.
2.00	9.1743	8.7236	8.2645	7.8431	7.4627	7.1174	6.7797	6.4725	6.1728	5.8997	
2.10	9.6330	9.1703	8.6777	8.2353	7.8358	7.4733	7.1186	6.7961	6.4814	6.1947	
2.20	10.0917	9.6700	9.0909	8.6275	8.2090	7.8292	7.4576	7.1197	6.7901	6.4897	
2.30	10.5505	10.0437	9.5041	9.0196	8.5821	8.1851	7.7966	7.4434	7.0988	6.7847	
2.40	11.0092	10.4804	9.9174	9.4118	8.9552	8.5410	8.1356	7.7670	7.4074	7.0797	
2.50	11.4679	10.9171	10.3306	9.8039	9.3284	8.8969	8.4746	8.0906	7.7161	7.3447	
2.60	11.9266	11.3537	10.7438	10.1961	9.7016	9.2527	8.8136	8.4142	8.0247	7.6697	
2.70	12.3854	11.7904	11.1570	10.5882	10.0747	9.6085	9.1526	8.7378	8.3334	7.9647	
2.80	12.8441	12.2271	11.5702	10.9804	10.4479	9.9644	9.4915	9.0615	8.6420	8.2596	
2.90	13.3028	12.6638	11.9834	11.3725	10.8209	10.3202	9.8305	9.3851	8.9507	8.5546	
3.00	13.7615	13.1004	12.3967	11.7647	11.1941	10.6761	10.1695	9.7087	9.2593	8.8496	
3.10	14.2202	13.5371	12.8099	12.1569	11.5672	11.0320	10.5085	10.0323	9.5680	9.1446	
3.20	14.6789	13.9738	13.2231	12.5490	11.9404	11.3879	10.8475	10.3559	9.8766	9.4396	
3.30	15.1376	14.4105	13.6363	12.9412	12.3135	11.7437	11.1864	10.6796	10.1852	9.7345	
3.40	15.5964	14.8472	14.0496	13.3333	12.6866	12.0996	11.5254	11.0033	10.4938	10.0295	
3.50	16.0550	15.2843	14.4628	13.7264	13.0598	12.4555	11.8644	11.3269	10.8024	10.3245	
3.60	16.5137	15.7205	14.8760	14.1176	13.4329	12.8114	12.2034	11.6505	11.1111	10.6195	
3.70	16.9724	16.1572	15.2892	14.5098	13.8060	13.1673	12.5424	11.9741	11.4197	10.9144	
3.80	17.4311	16.5939	15.7025	14.9020	14.1792	13.5231	12.8814	12.2977	11.7238	11.2094	
3.90	17.8899	17.0306	16.1157	15.2941	14.5523	13.8790	13.2204	12.6213	12.0370	11.5044	
4.00	18.3486	17.4673	16.5289	15.6862	14.9254	14.2349	13.5593	12.9450	12.3457	11.7994	

Double carbonatation adopted to obviate the redissolving of lime and impurities by excess of carbonic acid.—The reactions described hitherto explain what occurs in the juice up to the moment of

defecation and the saturation of the lime by carbonic acid. But if the saturation by carbonic acid be carried too far, a portion of the coloured carbonate of lime redissolves, re-embodying in the juice a portion of the precipitated impurities. Again, on the other hand, if the saturation be not carried far enough, juices very alkaline by lime are obtained, which are very difficult to concentrate.

With the view of obviating these two difficulties, *Perier* and *Possoz* conceived the very simple idea of separating the bright, very alkaline, supernatant juice from above the coloured calcareous precipitate, and continuing the saturation of this juice in another vessel, so as to complete it. They even added a little fresh lime in carrying out this second carbonatation, and thus rendered the process more effectual still, and thus was originated the process destined to completely supersede *Rousseau's*.

The modern filter press comes to the aid of the double carbonatation process.—It is right, however, to point out that, simultaneously with this new process, a new machine was given to the world, and which enabled the sludge to be separated mechanically from the clarified juice, namely the *Filter Press*, which will be described at the right time and place.

Here, however, we may interpolate a table showing the chronological development of carbonatation processes in France.

XXXIV.—METHODS OF DEFEICATION AND CARBONATATION, 1884–1885
TO 1899–1900.

Year.	Number of Factories using		
	Simple Defecation.	Single Carbonatation.	Double Carbonatation.
1884–85 . . .	42	28	379
1885–86 . . .	28	11	374
1886–87 . . .	15	7	369
1887–88 . . .	1	4	370
1888–89 . . .	1	3	376
1889–90	371
1890–91	377
1891–92	368
1892–93	368
1893–94	370
1894–95	367
1895–96	356
1896–97	358
1897–98	344
1898–99	344
1899–1900	339

Details of the double carbonatation process.—The double carbonatation process, as it was conceived by Possoz the chemist, Perier the engineer, and Cail the constructor, is carried out as follows:—

Liming.—Milk of lime is added to the *cold* juice as soon as it is extracted from the beet, *i.e.* immediately it comes from the diffusion battery. The juice should not remain exposed to the air without being limed, as it is apt to ferment (p. 121). The liming is generally done in a carbonatation pan, but, what is better still, sometimes in a special vessel provided with an agitator (Fig. 29). From 1.5 to 3 per cent. of lime reduced to milk of lime is added. (See Unger's table on p. 124, and further particulars on p. 121.)

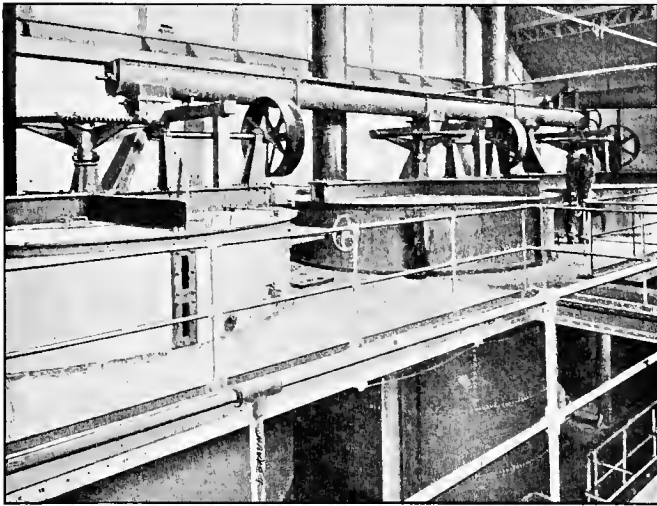


FIG. 29.—Special vessels for treating the beet juice with milk of lime (CAIL)

Injection of carbonic acid.—Froth prevention.—As soon as the milk of lime is added to the juice the carbonic acid is commenced to be passed through. A thick froth forms, which is kept down either with a fatty substance or a solution of camphoriferous oil in alcohol, which causes the balls of juice filled with gas to glide rapidly the one over the other and to split them rapidly, or with froth preventers to be described later on.

Application of heat.—When carbonatation has had a good start heat is applied by means of a steam coil which makes several convolutions round the pan, and the opening of the steam tap is so regulated that the juice reaches a temperature of 75° to

80° C. (167° to 176° F.) by the time carbonatation is nearly completed.

The near completion of the process is indicated by the disappearance of the froth and by the special colour which the liquid assumes. The workman then diminishes the injection of gas, so as not to go beyond the point at which he ought to stop.

The final point of the process, and signs by which it is recognised.—Moreover, this final point was easily ascertained in the following way. A little juice was taken in a big spoon, and examined in the light. When the carbonate of lime was seen to slowly separate from the juice, and to precipitate on the bottom of the spoon, whilst meandering over its surface, leaving a supernatant bright straw-yellow juice, the operation was finished. If the precipitate took too long in forming, the operation was not yet finished. If, on the other hand, the carbonate precipitated too quickly, and the juice was of a brown colour, the saturation point had been exceeded.

Chemical control.—At the present day the spoon test, the only test formerly in use, is controlled by chemical analysis. According to the nature of the beet, the amount of lime to be left in the juice at the end of the operation is fixed beforehand, say 0·1 to 0·2 per cent. The process is stopped when this point is exactly reached. The workman, in that case, has a small laboratory arranged on a table in front of his pans, which enables him very rapidly to test the amount of lime in the juice (see p. 147). Possoz, in fact, elaborated a system of analysis which was also very simple, but which analytical progress caused to be abandoned. Nevertheless, it is to him that the chemical exactitude of this first carbonatation is due.

Turning off the CO₂ gas.—When the exact point is reached the gas arrival tap is closed and the steam increased, so that the juice attains a temperature of about 95° C. (203° F.). Then the steam is turned off, and the pan immediately emptied into a vessel beneath.

The juice formerly remained in the vessel, into which it was decanted as described, until the muddy precipitates were collected at the bottom. The clear supernatant juice was drawn off through an elastic pipe, the head of which floated on the liquid, supported by a buoy. As soon as the juice commenced to come away turbid the decantation process was stopped, and the muddy residue was sent to the filter presses.

Filtration of liquor from first carbonatation.—Nowadays the turbid juice, as it comes from the carbonatation process, is immediately sent to the filter presses without deposition followed by decantation.

Beneficial results.—The result of this method of working is that the juices to be subjected to the second carbonatation are more clear and not nearly so turbid. This is a great advantage, as the mud of the first carbonatation contains the major portion of the impurities of the juice, and all the mud which escapes from the first carbonatation,

and passes into the second, is so much impurity, which will be

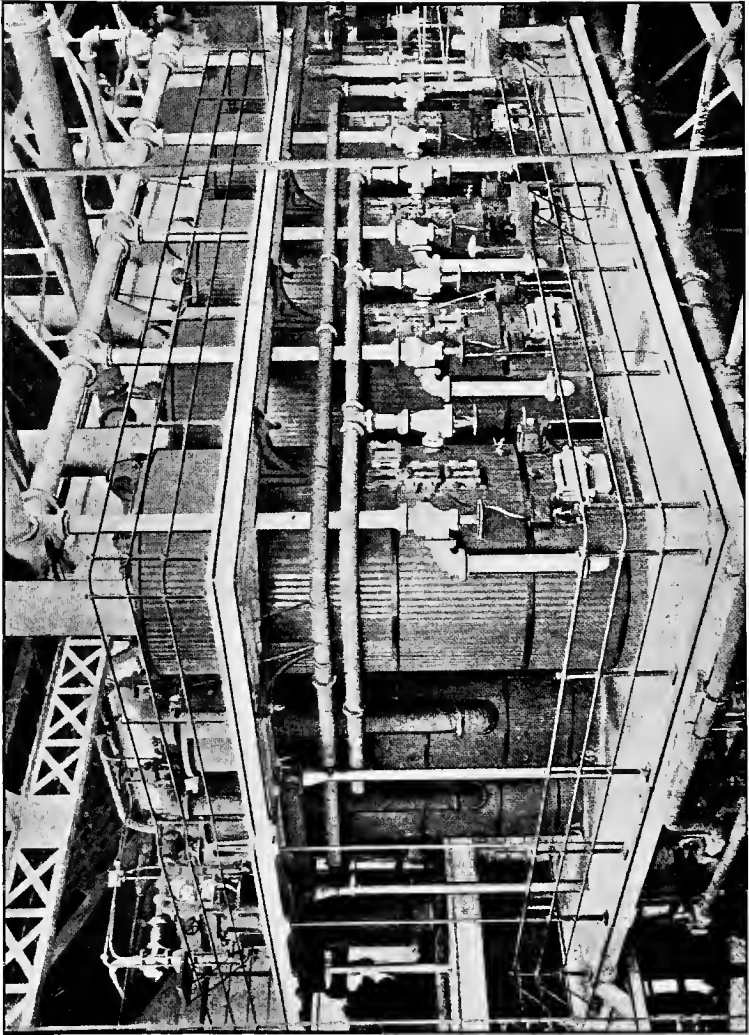


FIG. 80.—Intermittent carbonation tanks (C&I).

redissolved in the juice owing to the complete saturation about to be imparted to it.

The second stage of the double carbonatation process.—The clear juice of the first carbonatation is therefore sent on to the second carbonatation process. Lime, in the form of milk of lime, is added to the extent of two to ten-thousandths according to the nature of the beets. Gas is then injected to completely saturate the lime, the juice is brought to 100° C. (212° F.), and the operation is finished.

This turbid juice is immediately run into settling tanks, or passed on to the filter presses, as in the case of the first carbonatation juice. The limpid juice thus obtained is sufficiently pure to be evaporated and boiled down in the manner to be described in the sequel.

The cold system of carbonatation described above was especially necessary in the case of *pressed* juice.

Modifications in the carbonatation process due to introduction of the diffusion process.—Since the diffusion process became general in beet-sugar factories, certain alterations have been made in the working of the carbonatation process.

Diffusion juice more hot and pure than pressed juice.—As a matter of fact, the diffusion juice has already been raised to a rather high temperature in the diffusers, namely 75° to 80° C. (167° to 176° F.). The diffusion juice, again, owing to the method of its extraction, is purer than the pressed juice. Osmosis has, in fact, already kept back certain substances which would naturally enter with the juice into the carbonatation process, retaining, in fact, in the beet cell a large proportion of the albumenoid and pectic bodies, which the pulping machines and presses liberated in such a way that they passed into the juice. A large proportion of the impurities present in pressed juice are thus already eliminated in the diffusion juice.

The purity of the juice the cause of the abandonment of diffusion in the first instance.—Moreover, it was precisely because the diffusion juice is purer than pressed juice that Mathieu de Dombasle was forced to abandon his maceration process. The juice which he obtained by maceration could not be suitably defecated for want of albumen. Even when the juices of certain beets were treated by Rousseau's process, defecation was so difficult that it was often necessary to add blood, or white of egg, or even cow's milk, to form a scum or coagulum to envelop and carry down the impurities.

Many precautions indispensable with pressed juice superfluous in the case of diffusion juice.—Some of the precautions necessary with pressed juice, such as conducting the first carbonatation in the *cold*, are altogether unnecessary in the case of diffusion juices. Moreover, there are some advantages in working in the *hot*, as the pectic and albumenoid bodies, existing in smaller proportion, then coagulate better in presence of lime.

Liming, temperature, and proportions.—*Defective German practice.*—The diffusion juice is limed in a mixing vessel (Fig. 29), then heated to 80° C. (176° F.) in a special reheater, and enters into the

first carbonatation in the *hot*. In certain countries, and more especially in Germany, it has been thought unnecessary to use so much lime with diffusion juice as with pressed juice, and they have accordingly diminished the quantity. But, instead of limiting themselves to a proportionate reduction, they went to the opposite extreme, and did not add enough lime. The result of this was, that in Germany, instead of two saturations it has been necessary to make three, because the first and only important one was defective.

French methods.—In France, however, they have remained steadfast in the sound belief of adding plenty of lime in the beginning, and they thus confine themselves to the second carbonatation, which under the circumstances is sufficient. This is, no doubt, the right method of working.

Summary.—Such is the normal method of working the double carbonatation process. But as efforts have been made to find other and possibly more preferable processes, other defecating agents have been discovered, which we shall now pass in rapid review, dwelling more particularly on those which are used as aids to carbonatation, and which are sometimes used conjointly with it.

Carbonatation by means of baryta.—Baryta is the defecating agent *par excellence*; it is the first element of the series of alkaline earths,—baryta, strontia, and lime,—and is the most active. Strontia comes after it, and lime the last of the three, as a defecating agent. Lime, however, is preferred, on account of its abundance in nature, its easy conversion from carbonate into quicklime, and last, but not least, on account of its cheapness.

Baryta far too costly, and its preparation insanitary.—Baryta, however, is sufficiently abundant as sulphate (*barytes*), but the sulphate is only transformed into caustic baryta with great difficulty, and disengages such enormous volumes of sulphuretted hydrogen in the process that its manufacture is banished from all inhabited places. Manufacturers are thus thrown back on the less abundant carbonate of baryta, witherite, from which the caustic baryta which is used in sugar manufactories is made.

Baryta too costly by itself.—Attempts have been made to use baryta by itself, but its cost is too great, and the revivification of the carbonate contained in the sludge is not profitable enough.

A sufficiently cheap and effective auxiliary in the second stage.—At the present day baryta is sometimes used as an auxiliary to lime in the second carbonatation, having the advantage in that case of acting as a precipitant of all the impurities which the lime was unable to remove in the first process. For this purpose baryta is very useful without being too dear.

Carbonatation in the diffusers by soda crystals and barium hydrate.—Another process consists in adding to the diffusion 1 lb. of carbonate of soda per 1000 lbs. of beets, thus perfectly neutralising the juice;

then, as soon as the juice is drawn from the last diffuser, 1 lb. of crystallised baryta stirred up in water is run into the vessel. This forms an abundant precipitate, and the lime is finally added without troubling about the baryta. The carbonatation is effected in the usual way. This method, they say, likewise gives excellent results.

Fears as to the toxic dangers incidental to the use of baryta.—The danger attendant upon the use of baryta in sugar manufacture, on account of its poisonous nature, has been the subject of comment. This fear is, however, regarded by some as groundless, because baryta precipitates are so insoluble that not a trace of this alkaline earth remains in the saccharine juices. To this, however, it may be replied that objection was taken to the use of basic acetate of lead under similar circumstances. During the season 1899–1900 seventeen beet-sugar factories used baryta as a defecating agent.

Strontia is not used as a defecating agent.—Strontia is not used in defecation. We shall, however, see later on (p. 271) that strontia finds a considerable use in the treatment of molasses, and even forms the basis of quite a new method of extracting sugar from these residues.

XXXV.—SOLUBILITY OF STRONTIA, STRONTIUM HYDRATE, AND CAUSTIC BARYTA IN WATER.

Temperature.	Per Cent. SrO.	Per Cent. $\text{Sr}(\text{HO})_2 + 8\text{H}_2\text{O}$.	Per Cent. BaO.	Temperature.	Per Cent. SrO.	Per Cent. $\text{Sr}(\text{HO})_2 + 8\text{H}_2\text{O}$.	Per Cent. BaO.
0	0·35	0·90	1·50	55	2·54	6·52	14·71
5	0·41	1·05	1·75	60	3·03	7·77	18·76
10	0·48	1·23	2·22	65	3·62	9·29	24·67
15	0·57	1·46	2·89	70	4·35	11·16	31·90
20	0·68	1·74	3·48	75	5·30	13·60	56·85
25	0·82	2·10	4·19	80	6·56	16·83	90·77
30	1·00	2·57	5·00	85	9·00	23·09	...
35	1·22	3·13	6·17	90	12·00	30·78	...
40	1·48	3·80	7·36	95	15·15	38·86	...
45	1·78	4·57	9·12	100	18·60	47·71	...
50	2·13	5·46	11·75	101·2	19·40	49·75	...

THE MACHINERY AND PLANT USED IN TREATING THE JUICE

The measuring tank.—When the juice leaves the diffusers it runs into a cistern called the measuring or gauging cistern, in which the amount of juice drawn from the diffuser is measured, and it is this measuring which assures uniformity in the density of the liquid and the proper working of the battery itself. Measuring cisterns vary in

number and size in different countries. In France there is generally only one. Certain factories, however, have two—one which is being *emptied* whilst the other is being *filled*. This is a mistake, because it in no way forwards the work when the measuring tanks are well designed and arranged. All that is required is that the cistern should empty itself rapidly through a wide pipe. This renders the employment of a second one superfluous.

The special measuring tanks in use in Belgium.—In Belgium the duty is collected on the juice drawn from the battery, and the quantity is measured in this cistern. It accordingly is of a special shape. It is a tank resting on feet with levelling screws, in such a way that it is quite horizontal. It is filled to overflowing all round, so as to make sure of the juice drawn off. The liquid runs over into a gutter, which discharges into a close vessel. The cistern itself is closed by a lid, and by resistant metallic wire gauze. The closed vessel, the lid, the taps, are all under lock and key, and no one can touch them without the concurrence of the exciseman, who notes each time the density, which is taken by means of a special automatic test-glass, likewise under key. There is thus no possible means of fraud, and the manufacturer is exactly informed as to the amount of juice drawn off. In France the information upon this point is not precise, for, in spite of the float and indicator, if the workman does not examine these at the right moment there is no guarantee as to the regularity of the work. To remedy this, Horsin-Deon designed his automatic register.

HORSIN-DEON'S AUTOMATIC REGISTER

A cylinder rotated horizontally by clock-work carries a roll of paper divided into hours, quarter-hours, and fractions of five minutes; a pencil car attached to an arm bears directly upon the paper: this pencil, moved by a float, follows, and registers every change of level in the measuring tank.

It will thus be seen that this instrument records every change that takes place in the measuring tank, whether it be charging, discharging, or irregularities of the work of any kind. The lines traced from the lower to the upper part of the paper show the charging of the measuring tank; and, *vice versa*, those traced from above downward the discharging; a horizontal line indicates a stationary level. A counter records the total number of cells whose juice has been drawn off. M. Horsin-Deon has so arranged this apparatus that it may be located at a distance from the diffusion battery, preferably in the office or laboratory. In order that the work may be regular and the extractions uniform, two electric bells are connected with the apparatus, and indicate the proper moment to open or close the battery valves.

With this apparatus, where a complete record of the diffusion

work is automatically made, one can obtain absolute certainty that all orders from the laboratory or office have been strictly carried out, and that time has not been lost at certain hours of the night, only

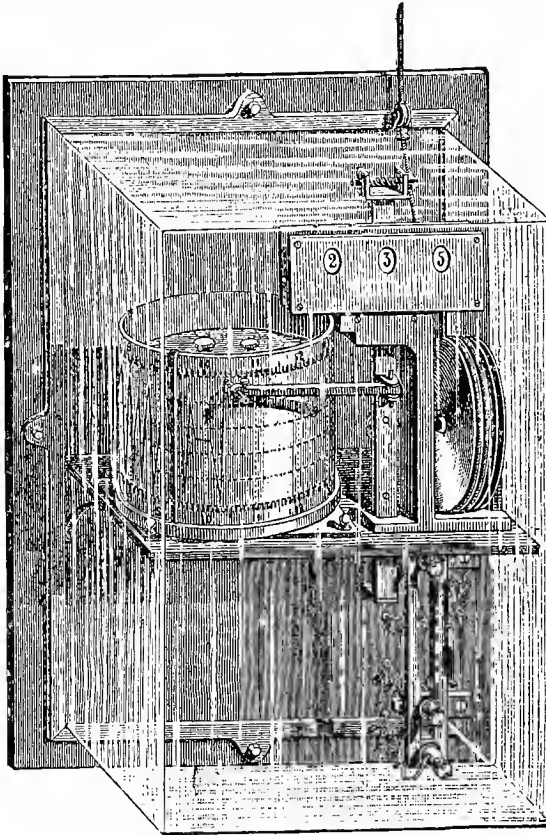


FIG. 31.—Horsin-Deon's automatic register.

to be made up by hurried work in the morning. The page is changed every 12 hours.

AUTOMATIC SAMPLING OF THE JUICE FROM THE DIFFUSERS

The easiest arrangement for automatically sampling the juice from the diffusers consists of a three-way valve, opened and shut by

the rise or fall of a float in the measuring tank. One of the openings of the valves communicates with a stand pipe extending above the greatest height to which the tank is ever filled, whilst the second opening connects the stand pipe with the bottle, in which the sample

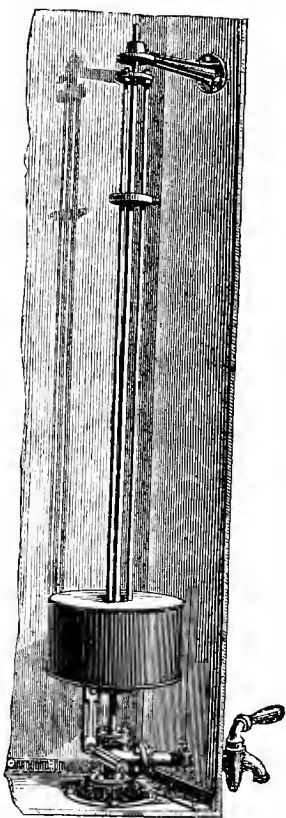


FIG. 32.—Apparatus for sampling automatically the juice from the diffusers.

is to be collected. The float is so adjusted with the stem of the valve that when the juice reaches a certain height it lifts and opens the valve, and places the stand pipe in communication with the tank. When the level in the tank falls, the opening from the stand pipe to the tank is closed, whilst at the same time that connecting the stand pipe and bottle is opened, and the juice which filled the tube passes into the bottle. This is repeated every time a cell of juice is drawn, and furnishes a sure and accurate sample. A certain amount of basic acetate of lead, proportional to that used in analytical work, is placed in the bottle to preserve the juice. Before analysis sufficient acetic acid is added to the sample, to give a decided acid reaction. An aliquot part of the sample is taken for polarisation and the determination of the glucose. Before making the glucose determination the lead should be precipitated and removed by filtration.

The opening in the three-way valve should be at least half an inch in diameter, to prevent clogging. The tube leading from the tank should also be provided with a fine strainer.

In order that the valve may work quickly enough to prevent placing the bottle in communication with both tube and tank at the same time, it should be fitted as follows:—

A section of the valve through the openings should exhibit a T-shaped groove, in order that a quarter-turn may suffice to connect the stand pipe with either the sample bottle or the measuring tank. The stem of the valve should be prolonged and fitted with a pinion $\frac{3}{8}$ inch in diameter, which in turn engages a spur-wheel 4 inches in diameter. The spur-wheel shaft is fitted with a drum 8 inches in diameter. The wire extending from the float makes a couple of turns round this

drum, and is then weighted. The float is so arranged that it has a rise or fall of about 1 inch.

The entire apparatus is provided with a suitable frame, and kept under lock and key. The delivery tube from the stand pipe extends nearly to the bottom of the sample bottle, in order that the stream of juice may thoroughly mix with the basic acetate of lead and with preceding charges. The dimensions of the gearing and drum given are such that a very slight change in the level of the juice in the measuring tank will open or close the valve.

EUGENE LANGEN'S AUTOMATIC REGISTER

The apparatus about to be described was designed by Eugene Langen of Cologne, a very prominent beet-sugar manufacturer, and

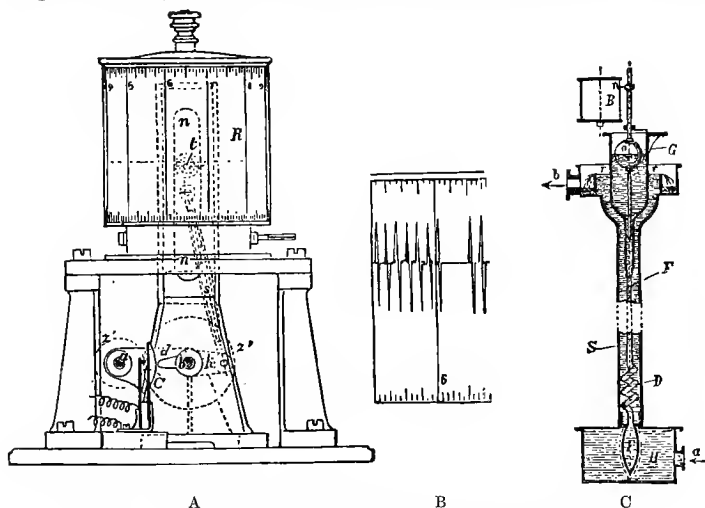


FIG. 33.—Eugene Langen's automatic register of the volume of juice drawn from the diffusers.

was constructed by Fischer & Strecht, Essen-am-Ruhr, Germany. It is designed not only to register the measurement of the juice, but also to determine its density.

The measurer of the volume of the juice drawn consists essentially of a copper cylinder containing six compartments, and is similar in its action to a gas-meter. The juice from the diffuser passes immediately into the measurer, thence to the carbonatation or clarification. The quantity of juice is indicated in cubic metres by means of the counter on a cylinder R. To accomplish this the counter com-

municates with the axis, which makes a complete turn for each diffuser of juice drawn. The axis a transmits its motion to the toothed wheels $z^1 z^2$ (variable at will) to the axis b , upon which is fixed a crank k and a projecting arm d . The crank, by means of the connecting rod s , raises or lowers the waggon t , carrying a pencil and travelling on the guides $n n$ in such a manner as to trace a diagram on the slowly revolving cylinder R , which is driven by clock-work. The arm d , touching the electric contact e , closes the circuit and rings an electric bell, which notifies the battery-man that he must close the juice valve. Fig. 30 B indicates the form of the diagram traced. The lines in the diagram, which are very nearly vertical, indicate the time required to discharge a diffuser of juice. The short horizontal lines show the length of times between discharges of juice and their height, above the centre line shows whether the correct amount of juice was drawn.

The automatic determination of the density of the juice depends upon the principle of communicating vessels. A column of juice of an invariable height counterbalances a column of water whose height is proportional to the density of the juice.

A portion of the juice measured by the meter passes through the small reservoir H , into a tube S , provided with an overflow at r . Inside the tube S is another tube $F D$, which terminates above in a funnel-shaped vessel and below in a flexible rubber bulb F . The interior of this tube, including the bulb, is filled with water, whose height is registered on cylinder b by means of a float carrying a pencil.

The variable temperatures of the juice have no influence on the apparatus, provided the column of water is of the same temperature as the juice surrounding. For this reason the tube F is spiral at the lower end. The specific gravity of the juice so obtained is reduced to the normal temperature, and the degrees Brix or Baumé noted. Langen has substituted a bundle of very fine copper tubes for the spiral, in order to more readily equalise the temperature of the juice and water.

Foam and mechanical impurities do not affect the accuracy of the apparatus. If this apparatus be used independent of the automatic measurer, a double ball valve should be employed to prevent wastage from the overflowing of the juice.

CARBONATATION TANKS

Liming in the measuring tanks.—The juice is sometimes limed in the measuring tanks, but chemical control then becomes impracticable. It is better to pump the juice to special tanks (Fig. 29).

The carbonatation tanks, their original form and defects.—The carbonatation tanks vary in size and shape. In France the first

tanks were square, wide, and shallow. The juice occupied a height of 20 to 25 inches in the tank. The carbonic acid gas was injected into the juice through perforated pipes forming a coil in the bottom of the tank.

Shallow versus deep carbonatation vessels.—This was a bad arrangement. It is much better in every way to pass the carbonic acid gas bubbles through a great depth of liquid, so as to utilise the gas to its fullest extent—to the best advantage.

The deep form of carbonatation vessels adopted in Austria.—With this end in view, in Austria they have for some time used very deep cylindrical vessels, containing a great depth of juice. French manufacturers have also begun to acknowledge that this is the better method of working. *Square deep tanks* (Fig. 30) are therefore now used for this purpose; the *square form* being preferred because the tanks can be fixed better one after another, so as to occupy less space. The depth of the juice is about 40 inches.

Prevention of frothing during carbonatation.—In order to get rid of the froth which is formed at the beginning of the carbonatation process, and which is so voluminous that it runs over the sides of the tank if a fatty body be not added, appliances called *froth eliminators* have been devised.

The various methods.—Froth eliminators are of two kinds. The first, known as Evrard's *emousseurs*, consist of a sheet of steam projected on the surface of the liquid, which breaks the froth. This is done by a pipe perforated with small holes placed horizontally above the liquid. Steam is run in at high pressure, which escapes with force through the whole of the small apertures in the pipe simultaneously.

Froth preventers are also used, based on the hydraulic turnstile principle, through which steam is passed.

These two appliances act well, but consume a great deal of steam. They have been generally replaced by mechanical froth preventers. These are shafts driven mechanically, and armed with blades which beat up the froth and destroy it.

Froth curbs like those of a soap pan.—But what acts better than froth preventers, and which is now adopted in all new factories, is to provide the tanks with a continuation or enlargement high enough to prevent the froth from overflowing. These enlargements, which are from 6 to 10 feet in height, are closed by a lid, itself surmounted by a very wide pipe passing through the roof (Fig. 30). All the annoyance and expense of froth preventers are thus got rid off. It was in Austria that Robert de Seelowitz first employed rational pans of great depth and enlargements of considerable height. Since then he has been copied everywhere.

The carbonic acid injection piping.—Inside each of the carbonatation tanks, resting upon the bottom of the tank, is a system of

perforated piping, either in the form of a square, a circle, or a star, for the injection of carbonic acid gas. The tanks are, moreover, heated internally by a steam coil, circulating right round the interior of the tank, or by the radiating heater (Fig. 36). They are emptied through an aperture inserted for the purpose in the lowest part of the inclined bottom, which is closed by a plug during working hours.

The tank is discharged either into a settling tank or into a mixing tank with an agitator in front of the pump, which pumps the carbonated juice to the filter presses.

The tanks used in the second stage.—The second carbonation tanks do not differ in any essential point from the first. However, they never are fitted with froth preventers, or if so they are very simple, because the juice, when heated a second time, froths much less than it did on the first occasion.

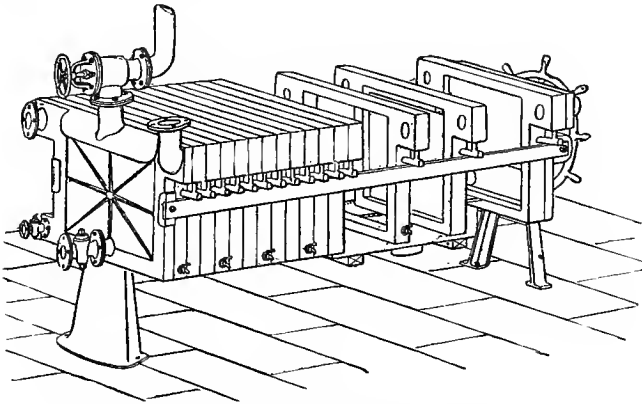


FIG. 34.—Horsin-Deon's continuous carbonator.

Continuous carbonation.—The carbonation tanks work intermittently, and would occasion a considerable loss of time if a numerous series of these pans were not kept at work, *two* at least being *idle*—*one* being *charged*, and the *other* being *emptied*.

Attempts were made for a long time to produce a system of continuous carbonation. With this end in view overflow tanks were constructed, and the gas injected in an opposite direction to that of the juice. But there is no absolute fixity of the percentage of the lime left in the juice treated. Again, the juice has been passed through pipes in an opposite direction to the gas, and the same result obtained, so that, after all the attempts, not one has survived in the manufactories. Looking at the matter from a different point of view to that in which it had been previously regarded, Horsin-Deon caused both the gas and the juice to pass

in the same direction, in such a manner that both the carbonic acid in the gas and the lime in the juice diminish simultaneously: working in this way when the appliance is adjusted and regulated, the small differences in the amount of lime used and in the richness in CO_2 of the gas produce but little alteration in the amount of lime in the juice.

Horsin-Deon compares the action of his appliance to that of Liebig's potash bulbs, in the form of a filter press, with a series of narrow chambers succeeding very wide ones, in such a way that in the latter the bubbling of the gas is highly divided. The limed juice and the gas flow in at one end of the apparatus, and flow out exhausted at the other.

When the apparatus gets soiled with mud, it is cleaned by dismantling it as in the case of a filter press. But these cleanings are not frequently required. Moreover, it occupies little space in comparison with the ordinary carbonatation pans.

Gibert's continuous carbonatation process in rectangular pans.—

This system comprises two rectangular pans, 1 metre to 1.2 metre in width by 3.5 metres in height, say 4 feet wide by 12 feet in height. These two pans are placed parallel, in the direction of the length, have each an inclined bottom, sloping in a contrary direction, so that the juice running through them in the direction of their length passes from the one to the other. In this 7 metres (say 24 feet) journey the juice meets, in each pan, special gas jets which distribute the gas in the mass of the juice. Again, as the level of the juice flowing away through a funnel is kept at a height of 2.5 metres, say $8\frac{1}{4}$ feet, it will be seen that the absorption is complete and the carbonatation, so to speak, instantaneous.

Precautions.—Certain precautions have to be taken in the working of continuous carbonatation. These are as follows:—

1. The liming should be regular; liming by powdered or lump lime is advisable.

2. The pressure of the juice as it flows into the tank should be constant.

The pan must not be fed by a pump, but preferably from a full cistern provided with a pressure regulator.

If these two conditions be fulfilled the carbonatation is effected regularly, and the carbonatation workman has nothing more to do than to regulate the gas-tap according to the results he gets on testing.

The advantages claimed for Gibert's system are space, economy, absolutely regular working, considerable output, perfect utilisation of the carbonic acid.

Maguin has applied Gibert's system to cylindrical pans, as shown in Fig. 35. In this arrangement the juice is fed into the second pan, either from the top or by means of a vertical pipe, starting at the bottom from the communication pipe, and ending at the top in an

elbow. In the second pan, at a level slightly higher than the level of the juice, or through the bottom even of this second pan, taps placed in the right position enable the bottoms of the pans to be flushed so

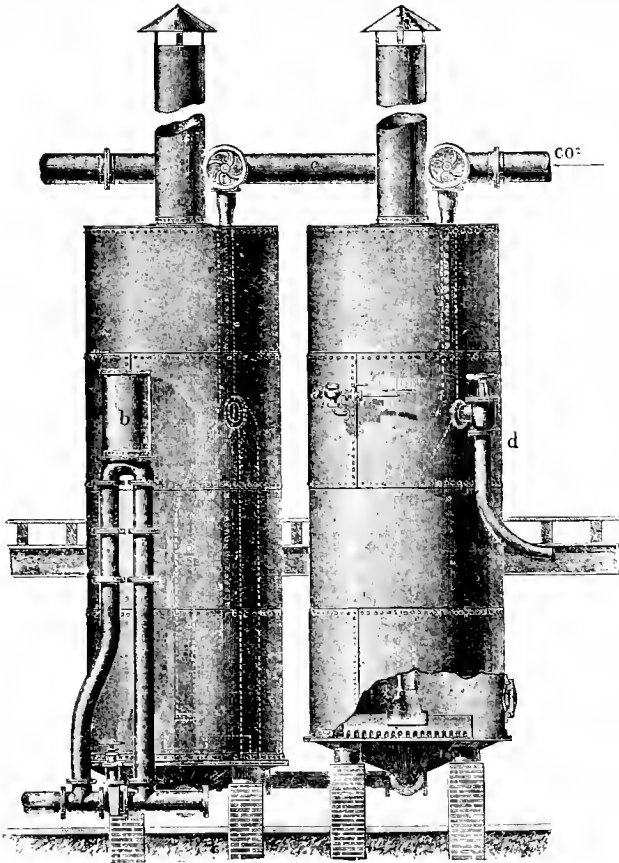


FIG. 35.—Gilbert's continuous carbonatation process (as applied to cylindrical pans by Maguin).

as to avoid any obstruction. A Desmet's froth preventer may be adapted to the first pan.

The radiating heater applied to carbonatation.—Not only does this apparatus (see p. 219) allow the carbonatation juice to be heated in the pan itself by the juice steam or the escape steam, but it also perfectly divides the juice and the gas, and produces a complete

emulsion from their interaction. With this end in view, the upper

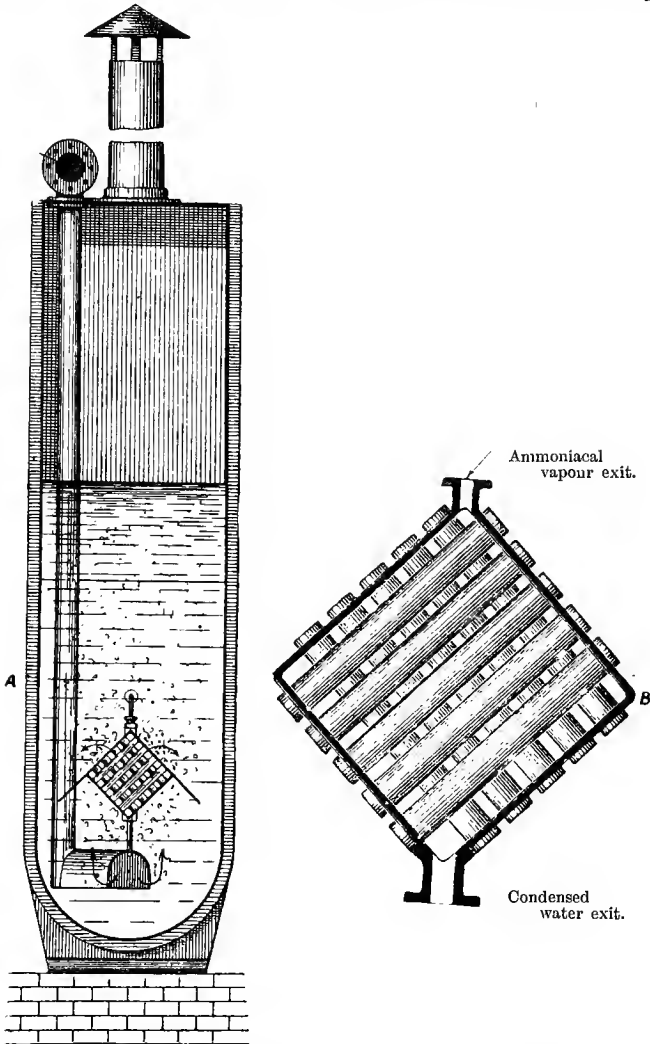


FIG. 36.—The radiating heater applied to a carbonatation pan.

sloped partitions of the radiating heater are prolonged by two wrought-iron baffles, which extend almost to the sides of the pan in

such a way as to force the gas to pass, in a very divided form, in an inverse direction, and thus form a very energetic circulation. Carbonatation is then accomplished very rapidly and under the best possible conditions.

Manufacture of carbonic acid and quicklime.—In Rousseau's process the carbonic acid was produced by burning charcoal in a special furnace. A pump drew the gas forward and pressed it into the limed juice. Sugar manufacturers had to buy lime or were forced to have a lime kiln in their factory.

The charcoal furnace superseded by the lime kiln.—But one day, very naïvely says Horsin-Deon, they were struck with the idea that burning lime in kilns produced carbonic acid. They then immediately set to work to extract the carbonic acid, which was evolved from the top of the lime kiln, since which time no other method was adopted, Rousseau's furnace being set to one side.

Peculiarities of a sugar factory lime kiln.—The lime kilns of a sugar factory differ but very little from those in general use, only their proportions are generally much larger, and these proportions correspond with the requirements of the factory. There is only one kiln as a rule to each factory, or at the most two, because it is difficult to regulate the working of several furnaces in identically the same way.

The lime kiln is on the continuous principle.—The lime kiln used in sugar factories is on the continuous principle, *i.e.* it is charged from the top and discharged from the bottom, in proportion as the lime is burnt, without, however, stopping the kiln burning for the purpose of discharging and recharging.

The method of heating the furnaces of the lime kiln.—Two methods of heating the furnaces are adopted. The one consists in having hearths underneath, on which a coke or wood fire is continuously kept burning. In the other method the coke which has to support the combustion is fed into the kiln with the lime, and there are no heating furnaces or hearths in which fuel alone is burnt.

The combination system of heating the kilns.—Sometimes, however, the two systems are combined, in which not only is the fuel mingled with the lime, but heating furnaces are also used.

The several methods of heating equally preferable.—If manufacturers are questioned, says Horsin-Deon, as to the best of the three systems, little satisfaction is got, each one praising the system which he adopts. From which it follows that they are equal. He prefers the second method as being the most simple.

The fuel should be free from sulphur and ash.—The coke used should be as free from sulphur and ash as possible, the sulphur giving rise to sulphurous acid, which militates against the successful working of the carbonatation process, whilst the ash introduces impurities into the lime which contaminate the juice.

Necessity for selecting a pure limestone.—The limestone itself ought likewise to be as pure as possible. It should be compact so as to produce lumps of well-burnt lime which do not split up when taken out of the furnace. *It should contain neither chlorides nor sulphates, magnesia nor clay.*

ANALYSIS OF LIMESTONE

1. *Moisture.*—Dry 5 grammes over the blue flame of an argand Bunsen. Loss multiplied by 20 equals moisture per cent.

2. *Insoluble silicious and organic matter.*—5 grammes are boiled in 100 c.c. water and 20 c.c. hydrochloric acid for 10 minutes; the insoluble is collected on a previously thoroughly dried filter, the weight of which has also been ascertained beforehand. The filter and its contents are washed and dried until of constant weight. This weight—minus that of the filter—multiplied by 20, equals amount per cent. of insoluble silicious matter plus organic matter.

3. *Insoluble silicious matter.*—The filter and its contents from 2 are ignited; the weight of the residue, multiplied by 20, equals per cent. insoluble silicious matter.

4. *Organic matter.*—The difference between the nett weights obtained in 2 and 3, multiplied by 20, gives the organic matter per cent.

5. *Sulphuric acid.*—An aliquot part after solution is brought to the boil, barium chloride added, the sulphuric acid estimated in the usual way and brought to per cent.

6. *Oxides, etc., of iron and alumina.*—An aliquot part of filtrate from 2 is oxidised by nitric acid and ammonium hydrate added, the precipitate washed, dried, and ignited in the usual way and brought to per cent.

7. *Lime* is estimated in the ordinary way in filtrate from 6 by precipitation with ammonia and ammonium oxalate and brought to per cent. of CaCO_3 .

8. *Magnesia* is estimated in filtrate from 7 by precipitation with hydric disodic phosphate, the precipitate filtered, washed with ammonia water, ignited and weighed, calculated to magnesium carbonate, and brought to per cent.

9. *Iron* is estimated in 10 grammes by dissolving in hydrochloric acid and titrating with decinormal solution of bichromate of potash; the result is calculated to per cent. of ferrous carbonate.

10. *Chlorine* is estimated by dissolving 5 grammes in nitric acid and treating with argentic nitrate in the usual way.

11. *Carbonic acid* is estimated by difference or in Schrotter's apparatus.

The limestone should contain a maximum of carbonate of lime and a minimum of moisture—10 per cent. at the most. After the limestone has been burnt it is necessary to ascertain the quality of the quicklime which it yields, and the proportion of inert ingredients

which it contains. This is done in a very simple but effective manner. A certain amount of sugar is dissolved in water, a weighed quantity of lime is added, and the whole stirred. The sugar dissolves the lime, and leaves a residue consisting of the inert substances. A more exact method could not be applied, as it is a repetition of what goes on in the factory.

In actual factory working it is necessary that the works laboratory should adopt the same methods of testing as the substances will be submitted to in the actual routine of factory operations. That is the best way of ascertaining the quality of the products, and of becoming acquainted with what goes on during the routine of factory work.

THE MANAGEMENT OF THE LIME KILNS AND PURIFICATION OF THE CARBONIC ACID

The limestone is broken into lumps the size of the two fists before being used to charge the kiln. The charging of the kilns and the speed of the gas-pump should be as regular as possible.

The amount of coke per ton of limestone burnt.—About 2 cwts. of coke are used for a ton of limestone. But the amount varies with (1) the good or bad construction of the kiln, and (2) the skill of the lime-burner. The top of the kiln is hermetically closed by a trap, or reversed cone, which forms a plug. Above is a hopper or funnel into which the coke and limestone are charged. When the charge is ready the trap is opened, the cone is lowered and immediately closed.

The rate of combustion is regulated by the speed of the pump.—Underneath the charging door a small tunnel is arranged in the masonry, opening to the outside, and from this aperture there branches off a pipe, in communication with a pump of sufficient force to aspirate all the gases formed. According to the speed of the pump the kiln works slowly or quickly. The speed of the pump must therefore be regulated so that the kiln works in a regular manner, *i.e.* with complete combustion of the fuel, and consequently *without the formation of carbonic oxide*, and without too much air in excess being carried through.

The percentage composition of the kiln gas.—The maximum theoretical richness of the gas obtained in this way is 44 per cent. of carbonic acid. But in actual practice the percentage varies from 25 to 30, never greater.

The manner in which the kiln is fed.—The limestone and the coke are raised to the top of the kiln by ingeniously constructed elevators, or by means of a simple rope and pulley. Baskets or trucks are used for this purpose.

Gas scrubbers and coolers.—Between the kiln and the pump the gas passes through one or several washers, which remove any ash or dust carried along in the train of the gas, and at the same time cool it,

for it is very hot, and as the work done by the pump is greater the cooler the gas, there is every advantage to be gained in cooling it well.

Description.—These washers or gas scrubbers consist of a vertical cylinder, divided into horizontal compartments by sheets of perforated wrought iron, to the number of four or five, according to the dimensions of the washer. The gas enters below and makes its exit from above. A current of water runs in an opposite direction from top to bottom, from which it escapes by a syphon. The perforated wrought-iron plates divide both water and gas, and cause a continual bubbling; moreover, a certain level of water is always maintained in the lower compartment, underneath which the carbonic acid enters

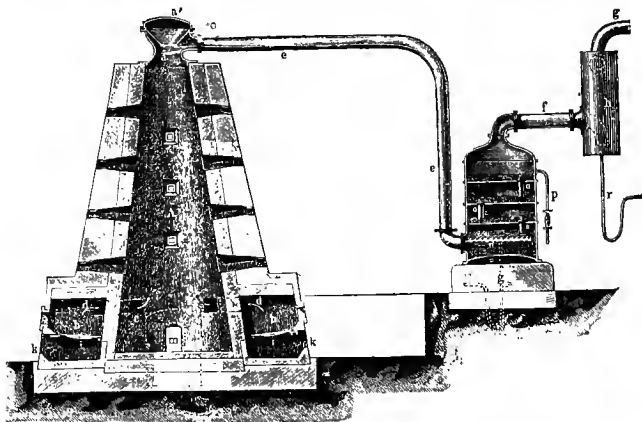


FIG. 37.—Beet-sugar works lime kiln and gas scrubber.

through a perforated pipe. Sometimes the upper compartments are charged with coke, so as to divide the gases and the water better.

Dust deposition chamber.—It is advisable to place a receptacle, in the form of a rather larger chamber, between the kiln and the scrubber, in which the greater amount of the dust may be deposited, and another chamber is placed after the scrubber, in which any water accidentally carried forward by the gas may be deposited. This latter chamber, moreover, serves another purpose, by acting as an elastic obstacle to the strokes of the piston, which without it would make themselves felt both on the scrubber and the kiln itself.

The gas-pump.—The gas-pump is an enormous cylinder, in which a piston moves, but not at too great a speed. This pump has no valves, but a plain drawer or cylindrical distribution. Its discharge pressure is made directly into the carbonators. But as some, or even the whole, of the tanks may be idle, the end of the pipe carries a

weighted valve, which rises and allows the gas to escape through the roof when the consumption by the carbonatation pans is insufficient.

Discharging the lime kilns and cooling the lime.—The lime is taken from underneath the kiln, or from the side, according to the model of kiln adopted. It is generally cold enough to be used at once. Sometimes, when it is discharged red-hot, it is necessary to cool it. But this only occurs with badly constructed kilns or abnormal working.

Slaking the lime.—The lime is transferred from the furnace to the tanks, in which it is slaked. This slaking consists in first moistening it with water, and then covering it with water until ebullition is over. When the mass is cold it is in the form of a thick paste.

Converting the slaked lime into milk of lime.—This paste is then run into mixers, which in a well-planned factory ought to be near the slaking tank. Sometimes, indeed, the slaking is done in the mixer itself. The lime mixers are cylindrical tanks (see Fig. 29), in which a vertical axis, provided with a rake of hanging chains, revolves. The chains, which rub along against the bottom of the tank, prevent any lime from accumulating there. The lime, in paste, is run into these tanks, along with water, and the rotation of the mixer produces a very intimate mixture called *milk of lime*.

Running the milk of lime through a sieve.—When the milk of lime is well made the contents of the mixers are run through a fine sieve, which separates the stones and the unburnt lumps, and a milk of lime, ready for immediate use, is now obtained.

The proper density of the milk of lime.—Matters are so arranged that the milk of lime marks 25° B. (sp. gr. 1.205), the density most favourable to its successful employment. That corresponds to about 25 per cent. of lime; the exact strength is, however, controlled by analysis. By taking the milk of lime as containing 25 per cent., it will be necessary to use 4 times more *milk of lime* than the actual quicklime judged as being necessary for the carbonatation. If, for instance, it be desired to use 2 per cent. of lime per hectolitre of juice, 8 litres of milk of lime are used, or 8 British imperial gallons per 100 gallons (see Unger's table, p. 124).

Gauging the milk of lime.—The quantity of milk of lime added to the juice is gauged by a measuring tank, placed above the juice mixer, and the carbonatation tank, which the workman fills and empties by means of taps and special piping leading from the milk of lime reservoir, in which an agitator is constantly at work to keep the strength constant.

The necessity for care in connection with the whole liming process.—All the work connected with the lime ought to be well managed and conducted, because on it depends the success of the carbonatation process. It is therefore one of the most important adjuncts of the factory which demands the greatest attention.

THE TITRATION OF THE LIMED JUICE

In a well-equipped factory, in which the chemical control of the manufacture is carried out in a systematic manner, the amount of lime in the juice is determined previous to carbonatation. But, in any case, the lime is always estimated at the end of the operation, both in the first and second carbonatation. As the determination has not only to be exact but rapid, the test is a volumetric one. The operation is performed on the spot. In front of the carbonatation tanks is a small bench, on which all the apparatus required for the test are fixed, and the workman is guided by his results in deciding when the operation in any given tank is finished. The apparatus may vary slightly in different factories.

The principal tool is a graduated burette or alkalimeter, fitted with a clip or stopcock, and containing the test acid. This test acid consists of sulphuric acid, diluted to such an extent that 1 c.c. = 0.001 of lime in the juice. A small quantity of the juice to be tested is filtered, and a known volume is measured out from it by means of a graduated glass on the vessel which is run into a test glass. The test acid is run from the burette into the liquid, drop by drop, until the liquid is neutral. The number of c.c. used indicates the number of thousandth parts of lime contained in the juice.

Litmus may be used as an indicator, but it is not quite sensitive enough for traces of lime; moreover, its colour is badly seen by night light. It is needless to remind the reader that litmus is blue in the presence of alkalies, and red in the presence of acids, and purple when neutral. Phenol-phthalein may also be used as an indicator; it is colourless in the presence of acids, but purplish-red in the presence of alkalies. A few drops of a neutral alcoholic solution of phenol-phthalein are run into the liquor, which is immediately coloured red, and the acid then run in, with constant stirring; and when the liquor being tested becomes colourless the operation is finished. Rosolic acid is also used as an indicator, in which case the liquid is changed from red to pale yellow. It would appear to give better results than phenol-phthalein. But perhaps the best results of any are to be obtained from the use of methyl orange as an indicator. Alkalies turn it yellow, acids bright red. Only enough is used to tint the solution the palest of straw-yellows. When neutral it passes almost instantaneously to a rose. In titrations far too much indicator is generally used. Only the smallest quantity of the coal-tar colours named is required. In the case of phenol-phthalein it is well to bear in mind that ordinary methylated spirit is slightly acid. Sometimes the test acid itself is coloured with the indicator, so that each drop gives its coloured reaction. The burettes are fixed by stands of different shape, and always include a rapid system of filling to the mark without making several attempts. Sometimes the test acid stock

bottle serves as a support to the burette, so as to reduce the apparatus on the bench to a minimum.

In fact, these simple tests are easily made by the carbonatation workmen, and tell them the point at which to stop. Carbonatation has thus become a regulated scientific operation, which is a great step in advance from the time they used to trust to rule-of-thumb work.

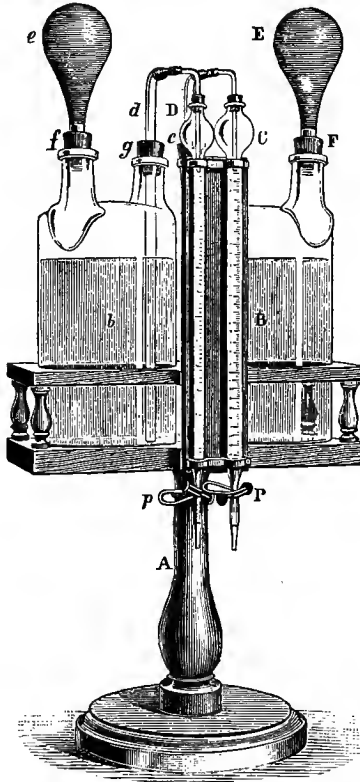


FIG. 38.—Sidersky's twin calcimetric burettes.

In order to facilitate the determination of the testing of the limed juice, whether in the sugar-works laboratory or in the works itself in front of the carbonatation pans, Sidersky has combined the calcimetric apparatus into a twin burette.

A is a revolving stand on foot, carrying a rectangular frame in which are placed two Woolf's bottles B and b, with two mouths. This frame is surmounted by a stand, on which are fixed the two

Mohr's burettes C and c, the upper parts of which end in bulbs. Each of the Woolf's bottles B and b has its mouth closed by india-rubber corks, each pierced with a hole. Through each of the corks F and f a tube attached to an indiarubber bulb E and e passes. The corks g are connected with the tubes D and d, which dip into the liquid and which communicate by means of small tubes bent at right angles and drawn out to a point with the burettes C and c. The burettes are closed by Mohr's clips P and p.

The bottle B and the burette C contain the alkalimetric liquid, the flask b and the burette c contain the soap test. The bottles B and b are filled by taking out the corks F and f connected with the bulbs E and e, which for the nonce are replaced by funnels. When the bottles are filled the corks are reinserted, care being taken to see that they are tight. By compressing the bulbs E and e the corresponding liquids ascend through D and d, and run into the corresponding burettes C and c. When the pressure is stopped the D d form a syphon and draw the excess of liquid from above the zero. The air of the burettes C c escapes through small holes made in the corks, closing the bulbs surmounting the burettes, thus avoiding any splashing of liquid.

The burettes are graduated so as to show directly the percentage of lime expressed in grammes per hectolitre of juice. The alkalimetric test is made by Le Docte's solution* working on 25 c.c. The total lime in the juice is determined by means of Boutron and Boudet's normal soap-test solution working on 40 c.c. of liquid in the usual way. The alkalinity of the carbonated juice, which is commonly determined as above, is generally expressed as quicklime (CaO), although very often the alkalinity may be partially due to fixed alkalies or to ammonia resulting from the decomposition of organic matter by lime. No practical method has yet been discovered of estimating the free lime, but this is of no great concern to the manufacturer. What he wants to know is the *alkalinity* on the one hand and the *total lime* on the other, both expressed in the same way as CaO, so as to compare the two tests, the relation of which to one another is of great importance in the carrying out of the second carbonatation.

The total lime may be estimated by Clark's soap test as modified by Boutron and Boudet. For proper chemical control each alkalimetric test ought to be followed by a "hardness" test, so as to see whether there are calcic salts in the juice or the syrup. This is not, however, done in actual practice, a few isolated "hardness" tests made in the laboratory being regarded as sufficient, whilst the alkalinity is being determined almost continuously. The carbonic acid used in carbonatation should contain a maximum amount of real carbonic acid, and neither carbonic oxide nor sulphurous acid.

* Dilute 4.38 grammes H_2SO_4 or 893 c.c. of normal acid to 1 litre. Working on 25 c.c. of juice, each c.c. = 0.1 gramme CaO per litre.

The percentage of carbonic acid in the gas from the lime kilns is estimated in the ordinary way. The gas is collected in a tube graduated into 100 c.c. or any other equal divisions, then a piece of caustic potash is introduced into the water. The whole is shaken until absorption ceases. The volume of gas which has disappeared gives the percentage of carbonic acid. This figure may vary from 20 to 30.

Special apparatus have been invented for executing this estimation. That of Possoz is the oldest; it consists of a large test-tube on foot, standing in a box in the shape of a cupboard. The absorption potash is in solution in a bottle, the contents of which are run forwards and backwards into a test-tube. The manipulation is therefore more simple than in the preceding method, and involves no risk to the operator's hands, which, in spite of all precautions, the potash attacks.

Orsat's gas analysis apparatus is well known, and it should form part of the equipment of a sugar-works laboratory in the same way as it does of all other well-equipped laboratories.

Determination of the purity of the carbonated juice.—Although the "purity" in this case, as in all others, is only apparent, it should be determined with great care. The carbonated juice contains about 10 per cent. of dissolved substances, so that a difference of 0·1 in the dissolved matter may lead to an error of a whole degree in the purity, —*e.g.* a carbonated juice with 10 per cent. of dissolved matter and 8·5 per cent. of sugar will have a purity of $\frac{850}{10} = 85$. The same juice, with 10·1 per cent. of dissolved matter, will have a purity of $\frac{850}{10\cdot1} = 84\cdot1$; again, if there be only 9·9 per cent. of dissolved matter, the purity will be $\frac{850}{9\cdot9} = 85\cdot9$. It is therefore necessary to determine the dissolved matter to the first decimal point at least, by which method only can the purity be determined in whole degrees.

NEUMANN'S STONE ELIMINATOR FOR SLUDGE OR MILK OF LIME PUMPS

This machine (Fig. 39) is fixed on the aspiration pipe of the sludge or milk of lime pumps, so as to mechanically separate the juice from suspended matter, sand, stones, and unburnt lumps, which have such a disastrous effect on the pump, pistons, cylinders, and valves, without counting the obstruction to the channels of the filter-press frames.

The juice enters, by an inclined tubulure, into the bottom of the machine, where it finds itself in rather a large space, in which heavy

bodies, sand and stones, are deposited, then, rising up the machine, it encounters a revolving sieve, with conical holes, through which it passes, freeing itself from any turbidity and suspended matters, after which it passes to a pump through the upper tubulure. The

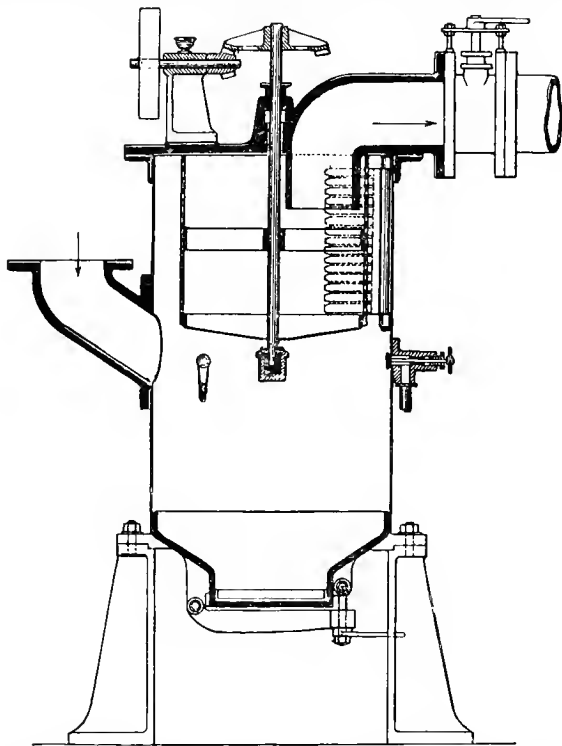


FIG. 39.—Neumann's stone eliminator for sludge or milk of lime pumps.

revolving sieve is constantly rubbed by a fixed scrubber, which removes the impurities adhering to the sieve. By a special arrangement it is possible to ascertain from the outside the height of the impurities inside the machine, and therefore when it is necessary to empty it, an operation which can be done very rapidly.

CHAPTER VI

THE EXHAUSTION OF THE SLUDGE—FILTRATION OF THE TURBID JUICES THROUGH FILTER PRESSES—BONE-BLACK FILTERS—MECHANICAL FILTERS—THE WASHING AND REVIVIFYING OF ANIMAL CHARCOAL—ANALYSIS OF ANIMAL CHARCOAL

THE EXHAUSTION OF THE SLUDGE AND CLARIFICATION OF THE TURBID JUICE

DIFFICULTIES in early days of separating the lime sludge precipitate from the mother-liquor.—The separation of the calcareous organic precipitate from the mother-liquor, after defecation and saturation, was for a long time a difficult problem. The sludge or defecation deposits are greasy, and do not filter through paper. Moreover, mechanical filters were badly adapted for the purpose. On the other hand, carbonatation, which produces a bulky deposit of sludge, could not be thought of, if mechanical filters were not available. Those interested were revolving in a pernicious circle, which for a long time retarded all inventions dealing with improvements in the purification of the juice. In the sugar refinery use had been made of bag filters, and efforts were made to utilise them for this purpose; but filtration in this manner took far too long.

The history of the filter press.—Howard thought of pressing the filters between frames, but his idea was premature. It was not until about 1850 and 1856 that Teedham and James Kite constructed wooden apparatus capable of working somewhat efficiently. Finally, Danek replaced wood by cast iron. Trinks improved existing filters; and from 1864, filter presses became quite efficient under the hands of Hoekner, Roetger, Durieux, etc.

Simultaneous introduction of carbonatation and filter press.—It will be seen that the real filter press was born simultaneously with Rousseau's and the Perier-Possoz carbonatation process, the one reciprocally assisting the other in their simultaneous birth.

The similarity in the filter presses of different present-day makers.—Filter presses are now made by all engineering firms almost on the same principles and in the same sizes. There are two models,

the largest of which, called originally the monster filter press, is becoming more and more generally used.

The general principle on which filter presses are constructed.—The principle on which these filter presses work is as follows. Suppose, for instance, a square cloth bag pressed on its four sides in a double armature or frame which prevents it from being deformed. The muddy liquid is forced into this bag under a pressure of two to three atmospheres. As the bag is held on both faces by perforated plates at a determined distance, the liquid passes through the holes, and the calcareous mud remains in the interior, forming a cake of the same thickness as that of the distance between the perforated plates.

The number of frames in a filter press.—From twenty-five to thirty, and even fifty, of these double frames are coupled together in the same appliance, thus forming the same number of bags into which the liquid is forced, and a filter press is thus obtained with a large filtering surface in a small space.

The ways and means of communication between the bags.—All the bags are in communication with each other. The variations of different makes are simply variations in the method of communication between the bags.

Danek's filter presses were constructed as follows. The bags communicated with each other on the top. With this object in view, all the frames had a wide hole in the centre of the top part. The cloths had a hole in front of these apertures, then a frame carrying a sheet of perforated iron was fixed in the pile, then a cloth, then an empty frame, then a cloth, then at last a frame with its perforated iron plate. The hole of the empty frame communicated by smaller holes with the interior, and it was in that way the muddy liquid entered and the cake became lodged in the same frame.

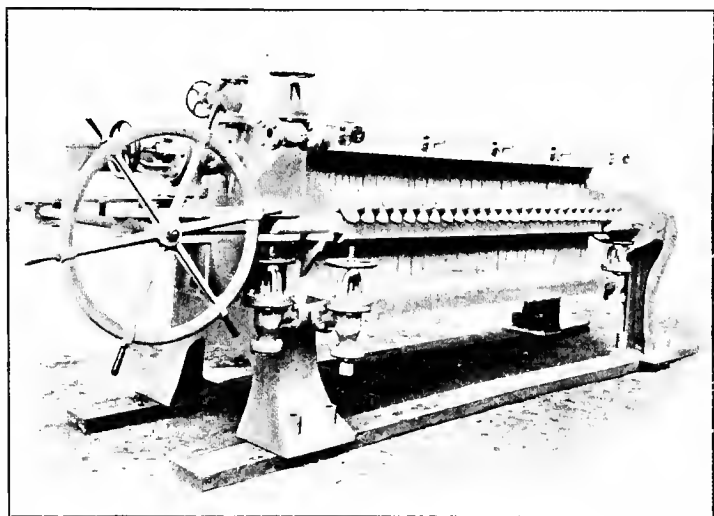
The frames with perforated plates carried, moreover, one of these plates on each of its two faces in such a way that it acted at the same time as the last frame of one bag and the first frame of the other. The fixed upright of the battery acted as the first frame, with perforated plate, and the last frame completing the battery also carried a plate; each of these frames was provided on both right and left sides with lugs, which rested on two horizontal pieces of iron, and the pressure of a strong screw kept everything in place.

Trinks did away with the intermediate frame by giving to the frames which carry the perforated plate a concave form, and then the hole for the muddy juice was made in the centre of the plate and the cloths. The latter made a tight joint with the frames by means of large screws, through the centre of which the mud escapes. The other parts of the press remained as before.

Filter presses on the Dehne principle.—The filter press invented by Dehne is the model most in vogue at the present time; and the

greater number of filter presses in actual use, not only in the sugar industry but also in every other industry in which filtration on the large scale is a prominent feature, are simply imitations of Dehne's type.

The principle on which the Dehne filter press acts.—Dehne's filter press is constructed on the same principle as Danek's, only all the frames have on the top and the side a lug, in which there is a passage through which the muddy liquid passes. A canvas washer, the same thickness as the filter cloths, serves as a joint between the opposite lugs. Nowadays these joints are perfected by indiarubber washers sunk into the cast iron.



[FIG. 40.—Beet-juice filter press (CAIL).

Size of plates.—(a) *The smaller filter press.*—The plates of the small filter presses used in the beet-sugar industry are 24 to 28 inches square. They run about twenty-five to thirty in a press, and hold about 10 cwts. of sludge.

(b) *The larger filter press.*—The plates of the larger-sized presses are 40 inches square, and form about eighty compartments. In fact, double filter presses have been built, more especially in Austria, with 160 to 200 compartments. But then, these are neither more nor less than two filter presses joined together end to end.

The tightening up of these monster filter presses (Fig. 40), instead of being simply done by a screw, which would not be powerful enough,

is effected either by hydraulic pressure or by a system of double-lever arms acting through the agency of a movable screw.

The function of the montejus in connection with the filter press.—The pressure of the sludge in the filter presses is effected by a montejus or by means of suitable pumps. The montejus is a stout cylindrical vessel into which the mud is run. A pipe in communication with the filter press leads down to the bottom of the vessel. When the cylinder is full, steam is applied through a tap in the top part of the vessel. The pressure of the steam causes the mud to rise up through the pipe and thus to fill the filter press. The whole of the contents of the vessel can in this way be expelled and replaced by steam. When that has been done the steam valve is closed, and a small side tap blow-off cock opened, through which all the steam contained in the cylinder is in its turn expelled. The montejus is again filled with mud, and the process repeated. By using two *montejus* the work of the filter presses can be carried on continuously and uninterruptedly.

Montejus superseded by pumps.—The great drawback to the use of montejus is the enormous amount of steam consumed in keeping them at work. They have therefore been replaced by pumps. But the difficulty with pumps is in maintaining a regular pressure in the filter presses, which must not go beyond a certain limit.

Accommodating or regulating the pump pressure to that required for satisfactory working of the filter press.—Two methods are adopted to accomplish the end in view. (1) The first method consists in constructing the pump in such a way that the ratio between the steam cylinder and the pump cylinder should be such that the machine becomes inactive when the pressure behind the pistons of the pump reaches the desired degree. (2) The second method consists in using a sensitive regulator, which shuts off the steam when the pressure in the filter press is as great as desired.

As a matter of fact, in well-constructed machines a combination of both methods is adopted. Moreover, the pump is double. There are thus two steam cylinders, and the same shaft serves for two couples, which are geared to this shaft in such a way that when one of the pumps is at the point of exhaustion the other is in full force. In this way the pump slows down, but starts again of its own accord as soon as the pressure lowers in the filter press.

Washing the sludge.—This is one of the most important points in the working of the filter presses. In fact, the cakes are wet with juice still containing much sugar, which it is, for economical reasons, necessary to remove as far as practicable. There are two methods of washing the cakes—

1. Washing the cakes in the filter press.
 2. Maceration of the cakes with water, and subsequent filtration.
1. *The first method fails by forming channels.*—The first method is

that most often used. Generally, water under pressure is run into the space, through which the juice flows out during the first part of the process. All the frames, therefore, have taps underneath. In the beginning all those corresponding with the sludge frames are closed, and the others are opened. During washing, the opposite course is adopted. To prevent mistakes, the keys of these taps are at different heights. But this method only partially eliminates the soluble matter. It consists, in fact, of only one water course, which simply conveys the water into the cakes, and then out by the tap in the adjoining plate. It will therefore be readily seen that the cake can only be but imperfectly washed, and, moreover, there is undoubtedly a great tendency for the water to form channels in the cakes and not to penetrate into and wash the other portions.

In Dehne's patent thorough extraction system.—After the formation of the cakes the taps are all closed. The water is now forced, under a suitable pressure—preferably a somewhat higher pressure than the one at which the press has been filled—into a channel, and is distributed over the filtering surface of the alternate plates, one having a channel to the inside, the next none. It is now forced simultaneously over the whole surface of the cake, to penetrate the cake, the next cloth to collect behind the filter cloth of the next plate, and finds its exit on the top of that plate, into the united efflux wash channel of every alternate plate. The air is allowed to escape through the channel, the valve of which must be closed as soon as the matter comes out. It is claimed, that the arrangement of these channels compels the fresh water to penetrate every part of the cake horizontally, and to take with it every particle of soluble matter.

In most instances the quantity of water to be forced into the filter press is decided upon beforehand. Sometimes washing is continued until a sample of the wash-water only shows 1° on the hydrometer. It depends on the routine and requirements of the factory. The wash-water goes to the second carbonatation. Sometimes the last portions of the wash-water are used to slake the lime.

Extreme washing unprofitable.—The sludge remaining in the filter presses after the filtration of the carbonated juice should only contain mere traces of sugar. A known weight is taken, washed repeatedly with boiling water, and the wash-water filtered, carbonated, again filtered, the proper quantity measured and polarised. One should not expect to extract all the sugar from the sludge, as that would involve washing beyond all measure, resulting in enormous volumes of very weak saccharine liquors, which it would never pay to concentrate, the working expenses being far greater than the values of the sugar recovered.

Mashing or maceration of the sludge.—The second method of

washing the sludge consists in mashing the sludge as it comes from the unwashed filter presses of the first carbonatation in a reservoir, in the bottom of which a stirrer revolves. Water is added, and when mixture is complete the whole is run into a fresh battery of filter presses. Sometimes this mashing is done in the unfiltered juice from the *second* carbonatation, so that the carbonate of lime, which filters much more easily than that of the *first* carbonatation, being added to the mud of the first, the cake is much drier and much more easy to wash afterwards in the filter press. As these mashing systems necessitate additional filter presses, and as the washing in the press itself is comparatively easy, except when the beets are bad or the carbonatation imperfect, the mashing process, although yielding very good results when it is well performed, is very seldom adopted.

The nature of the cake a key to good or bad working.—The emptying of the filter presses is performed by unscrewing the frames and letting the cakes of sludge fall between the feet of the framework, and then scraping the cloths. When the cake is fairly dry and well washed this operation is easy, and entails but little labour. But when carbonatation has been badly done the mud is tacky, washes badly, and sticks to the cloths. An inspection of the cakes shows, by itself alone, whether the factory is working well, or the reverse.

The floor on which the filter presses are located is generally above another floor, on which the trucks of a small tramway run. In this way, by opening a trap-door between the supports of the filter press, the cakes drop into the trucks which convey them to the sludge heap.

The sludge heap is therefore one of the necessary adjuncts to every beet-sugar factory. The trucks are emptied on to the heap in the following way:—

Matters are so arranged that the floor on which the trucks run is 12 to 16 feet above the ground, or more if practicable. The line of rails is prolonged outside the factory by a soundly supported staging or scaffolding, extending pretty far into the centre of a large courtyard. The waggons are discharged from the top of the stage, and the mud gradually forms a mound at the foot of the stage. The dimensions of the space available for this purpose ought to be calculated beforehand, so that all the sludge made during the season may be run on to it. The heap smells badly, owing to its undergoing butyric fermentation. This forms one of the nuisances of the neighbourhood of a beet-sugar factory, which should not be allowed to accumulate if at all possible.

The mud obtained from the filter press of beet-sugar factories, in the manner described, constitutes a valuable mechanical manure for certain heavy clay soils, to which it imparts lightness. But, besides

being a mechanical manure, this substance is also a plant-food—a manure in the true sense of the word. It contains—

XXXVI.—ANALYSIS OF BEET-SUGAR SLUDGE.

	Per Cent.
Nitrogen	3 to 4
Phosphates	4 to 5
Organic Matter	8 to 10
Lime	25

Unfortunately this waste product is very bulky and cumbersome, and it is not required in all beet-producing districts for manurial purposes. In any case it is a long time in being got rid of, and the sugar manufacturer must wait until the autumn before the farmer takes delivery of it. An outlet for substances like this, after drying, is often to be had by selling it to the makers of chemical manures, who utilise them as driers, or make weights for their freshly made manure. Unfortunately, in this case the sludge abounds in carbonate of lime, which would convert the “soluble” phosphate into “insoluble” phosphate. The use of beet-sugar sludge is not therefore permissible for this purpose, but that does not detract from its own manurial value in any way. Moreover, the manure manufacturer can with profit send it up the cups and treat it with acid in the usual way.

ESTIMATION OF THE SUGAR LEFT AND LOST IN THE SLUDGE

The following are the details of the analytical process. The analysis is made in duplicate by two different processes.

1. *Total sugar*.—The normal weight is triturated in a mortar with water and exactly neutralised with acetic acid, a few drops of basic acetate of lead are added, the whole made up to the mark and filtered, and polarised as far as possible in a large tube.

2. *Sugar existing as sucrates*.—The above operation is repeated without neutralisation and without precipitation with basic acetate of lead. The polarisation results give the sugar existing as juice. Any difference between the results points to the presence of an insoluble sucrate.

Precautions.—1. *Sampling*.—The exact determination of the sugar lost in the sludge necessitates many precautions. The sample cake should correspond with the cakes of the fore, centre, and aft parts of the filter press, as well as with the different parts of each cake, because the sugar is not uniformly distributed throughout the cake. Moreover, a great number of small samples should be taken

from each filter press, and the whole mixed so as to get a fair average sample for analysis.

2. *Analysis*.—The analysis in itself is comparatively simple. Care, however, must be taken to triturate the sample with water, and to neutralise it exactly with a few drops of acetic acid, which can be easily ascertained by a drop of phenol-phthalein, otherwise there is the risk of decomposing certain compounds of lime with organic acids. These acids being dissolved by the liquid, might affect the polarisation results. The amount of sugar in the sludge being small, and the solution colourless, the polarisation is made in a 500-millimetre tube.

Total weight of sludge.—It is difficult to estimate the total amount of sludge produced. It is advisable to calculate it directly from the number of presses emptied, taking as a basis, from time to time, the weight of the contents of one or more presses. In default of more exact data, as the sludge contains about 25 per cent. of lime, an approximation may be made by multiplying the quicklime used in the carbonatation process by 4.

The juice from the filter presses requires filtration through bone black.—The bone-black filtration process and the chemistry of bone black will only be roughly outlined here; the subject will be more fully elaborated under Sugar Refining. However limpid the juice as it flows from the filter presses may appear, yet it is not quite free from suspended particles, the more so if the cloths be rather loose in texture. Moreover, it is very rare that there are not some of them slightly perished, either through age or because of holes of larger or smaller size. Again, when a filter press is set to work, the first runnings of the juice are always slightly cloudy. It follows that, as all these liquids run into the same receiver, the resultant fluid is not perfectly clear and bright, but more or less turbid.

Filtration necessary to obviate the bad effects of turbid juice on subsequent operations.—It has already been pointed out (p. 127) that any sludge particles brought in the train of the juice, from the first to the second carbonatation, are so much impurities re-entering the juice, owing to the fact that this sludge dissolves in the juice under the influence of the carbonic acid. Should the juice from the second stage of the double carbonatation be evaporated directly, if they be turbid, they soil and encrustate both pans and syrups. All these liquids therefore require filtration until perfectly bright and clear and altogether free from matters in suspension.

CHARCOAL FILTERS

History.—In the now obsolete defecation process the juices were alkaline, owing to the presence of lime, which was sometimes used in excess. They were also rather dark in colour. The lime was sub-

sequently injurious in the boiling, and also to the final crystallisation, and, owing to the resultant dark colour, it was impossible to obtain fine sugar at the finish of operations.

Figuier discovered and Derosné applied the superior decolorising properties of bone black.—Somewhere about 1800 the decolorising action of charcoal on liquids containing vegetable colours was discovered. In 1811 Figuié pointed out that animal charcoal was a much more powerful decolorising agent than wood charcoal. In 1812 Derosné applied this property of bone char to the refining of the saccharine liquids of sugar factories and refineries.

The liquors were originally boiled with char, albumen, etc., from

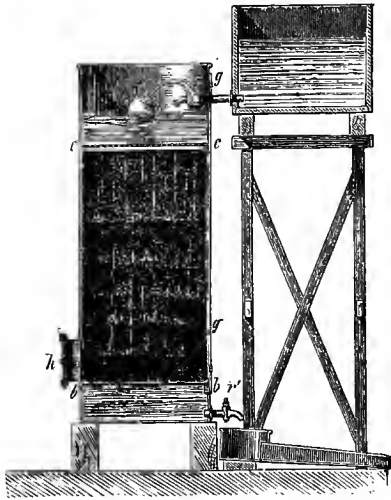


FIG. 41.—Dumont's open animal-charcoal filter.

which they were separated by pressure.—Up to 1828 finely powdered bone char was mixed with the liquid to be decolorised,—juice or syrup,—and it was boiled with albumen, blood, or white of egg. The albumen, in coagulating, was incorporated with the black. The whole was run into canvas bags and then pressed.

Filtration through cylindrical tanks packed internally with bone black (Dumont filters) obviates use of albumen.—But at this period, as a result of the researches of Payen, Dumont constructed char filters (Fig. 41) in the shape of a cylinder open at top, of from 16 to 20 feet in height; but instead of introducing char dust he used granulated charcoal. The juice passed through these filters was decolorised equally well as when mixed with the powder. The necessity of using albumen and bags

was thus obviated ; but not only so, the charcoal was left in such a condition that it was possible to revivify it.

Discovery of the revivification of bone char.—Payen found that, by reburning the char utilised in this manner, it regained its original bleaching properties.

The revivification of bone char opens up a new epoch in the history of the sugar industry.—From that time forward sugar factories and refineries were endowed with quite a practical method of purifying their juices, as the char could be used almost indefinitely, and the installation of Damont's system of filtration facilitated the rational use of the decolorising agent. Moreover, many forms of new and improved bone char revivifying furnaces came into being.

It constituted the first great successful step in advance which made further improvement possible.—We may safely say that the use of bone char constituted such a step in advance, such a real progress, that it was owing to it that the beet-sugar industry made its first great success, and that Rousseau's process became a practicable one.

The great affinity or attraction of bone char for mineral substances.—In addition to its decolorising properties, bone char is, moreover, endowed with a special affinity or attraction for mineral matter, which it seizes hold of and retains in its pores when their solutions are passed through it. In this way a solution of sugar, containing a little lime, filtered through granulated charcoal, issues from the filter free from lime, the latter having been absorbed by the char. The same absorption occurs with saline solutions, and a slight absorption in the case of sugar solutions.

Bone char a purifying agent as well as a decoloriser.—Consequently the bone-char filters not only act as decolorising agents, but they also act as purifying agents in the case of juices contaminated with mineral salts, and more especially organic salts. The bone char not only absorbs and retains suspended impurities, but dissolved saline substances in addition to colouring principles.

It will be readily appreciated, therefore, that animal-charcoal filtration was regarded, and justly so, as a valuable aid to the older methods of working, which, as may be readily conceived, were not by any means so exact as those in vogue at the present day. Its use therefore soon became quite general in all sugar factories and refineries, who used it quite regardless of cost, because by its use alone could a sufficiently clear and bright juice, prone to crystallise rapidly and with a big yield, be obtained with certainty.

The double carbonatation process obviated the necessity for so much bone-black filtration.—However, on the discovery of the Perier-Possoz system of chemical purification, it was found possible to lessen the enormous quantities of bone char used.

Its action then simply mechanical.—Moreover, in certain factories,

where carbonatation was well conducted, the charcoal filtration simply acted mechanically by arresting substances in suspension.

Bone-black filtration superseded by introduction of real mechanical filters.—Then mechanical filters, in the true sense of the term, came into use, and are so much improved at the present day that there is no further necessity for bone char, which has almost disappeared from the beet-sugar industry. Barely a few factories still use it in countries where the carbonatation process is not so well conducted as in France. The time, however, is drawing nigh when bone char will simply be a memory of the past, as many another process which has had its day in its time.

Its only use nowadays is in the manufacture of loaf-sugar in sugar refineries.—Nevertheless, its use is still continued in those refineries which make perfectly white loaf-sugar. It is practically only for this purpose that it is employed nowadays. Animal charcoal, or more familiarly “bone char” or “bone black,” is made by calcining the bones of animals without access of air. Long bones, such as the tibia and the femur, are preferred. Although both the bones of the horse as well as those of the sheep are used in charcoal burning, yet cattle bones are those which are most esteemed for this purpose. Boiled butchers’ bones are the best, therefore those which have been used for culinary purposes are preferred, the 10 per cent. of fat which they still contain being previously extracted by petroleum spirit.

Bones from glue factories from which the bulk of the ossein—constituting 33 per cent. by weight of the bone—has been extracted are very inferior and unsuitable, and should therefore be rejected. Spongy bones—the heads of the femur, for example—should also be set aside. Animal-charcoal makers, however, use a certain quantity, which they manage to pass with the others. These bones, previously ground, are introduced into perfectly cylindrical pots with a flat bottom, which are piled in a furnace, consisting of four walls without any other exit than a small air-hole, capable, moreover, of being regulated by a damper and a chimney.

A small wood fire is lighted under the first pots near the air-hole. The pots, becoming heated, give off gases, which take fire. From that time forward the furnace is heated from place to place by the combustion of the gas, which escapes successively from each of the pots. When the combustion is finished the furnace and its contents are allowed to cool, and the bones in the pot are then found retaining their original shape, but perfectly black. The black bones constitute the animal charcoal. The black bones are ground in crushers to lumps of the size of a filbert at the most. The char dust is sifted off and sold to the refineries, and nowadays to the boot-blackening manufacturers, and the large granules are used in sugar factories and refineries to pack the bone-black filters.

Animal charcoal contains roughly—

XXXVII.—ANALYSIS OF ANIMAL CHARCOAL.

	Per Cent.
Carbon	10 to 11
Tribasic phosphate of lime, $\text{Ca}_3\text{P}_2\text{O}_8$.	75 to 80
Carbonate of lime	8
Soluble salts
Insoluble salts

Owing to the presence of these soluble salts, it is necessary to wash new charcoal before use; unless this were done, they would spoil the sugar liquors by dissolving therein. Accordingly char which has been once revived is always better than new char.

Bone black is also made by distilling bones in retorts similar to those used in gas-works. In fact, this is the only rational way of making animal charcoal, the more so because at the present day the process is far from being a lucrative one; and if in the palmy days of the sugar industry the high prices obtained rendered it unnecessary to work up the residual products, it is not so now, when animal charcoal itself is a drug on the market. By distilling the bones in gas retorts, therefore, the residual products may be collected. First of all, there is given off an aqueous ammoniacal vapour, which condenses to an ammoniacal liquor, which is wrought up into sulphate of ammonia; then, again, a considerable amount of bone tar, or bone oil, which is again distilled for the production of naphtha, or is wrought for the manufacture of alizarine and aniline dyes, in the same way as coal tar, over which, in some instances, it possesses decided advantages in containing certain valuable colour-forming principles (*chromogenes*), which are either not present in coal tar or are so to a far less extent.

In the still in which the bone tar is distilled there is left a considerable residue of bone pitch, which is sold to varnish makers at about threepence per pound, and is used in the manufacture of Brunswick black and superblack Japan varnishes.

An animal-charcoal factory, therefore, which utilises its residuals to the fullest extent, is the only one which can at the present time ever hope to be a financial success.

There is a loss in the conversion of bones into animal charcoal

of about 30 per cent., that is to say, 15 tons of bones yield about 10 tons of char.

There are two kinds of animal-charcoal filters—open filters and closed filters. The open filters (Fig. 41) used in France in beet-sugar liquor decolorisation are from 10 to 16 feet in height, by 30 to 40 inches in diameter. The side of the cylinder, almost at the bottom, is pierced by a square manhole *h*, closed by a cast-iron door resting on a rubber joint, against which it is pressed by a screw. A perforated plate of wrought iron, *b b*, forms a false bottom.

Charging the animal-charcoal filters used in beet-sugar factories in France.—A coarse cloth is laid over the perforated false bottom described in the preceding paragraph, the manhole door is closed, and the cylinder charged with bone black from above. Another cloth is placed on the top of the black. The juice or syrup flows through a tap on to the cloth, which prevents the liquor from digging out channels in the bone black, and thus causes it to be evenly distributed all over its surface. The juice which has percolated through the char escapes from the bottom by an opening made below the perforated false bottom, and on the same diameter a screwed pipe is fixed to this opening by a nut and ascends vertically parallel with the cylinder, being bent into the form of a swan's neck at two-thirds the height of the cylinder. In this way the juice flows gently under a slight pressure, and the filter is always full. Moreover, the flow is regulated by a tap. The swan's neck (Fig. 82) moves freely round the axis of the pipe, so that the juice may flow into one of three parallel troughs, which run along the length of the row of filters—one of which is for juice, the second for syrup, and the third for wash-water.

Several filters are thus arranged in a row, according to the size of the factory—one filtering juice, another syrup, one being washed or empty whilst another is being filled.

The filter is emptied by running off the last dregs of the liquid through a tap in the bottom, whilst the dirty black is removed through the manhole in the side (*h*, Fig. 41; R, Fig. 82).

The closed charcoal filters used in beet-sugar manufacture in France.—Closed filters have the advantage over open ones of enabling the filtration process to be carried out under pressure. The cylinders are generally taller and narrower than the open ones; like the latter, they have a false bottom, a manhole for taking out the dirty char, and an exit pipe through which the clarified liquor is discharged. The top of the cylinder consists of a piece of cast iron, on which is a horizontal door, which is hermetically sealed like the one underneath; and a pipe, through which the liquid to be filtered is run into the cylinder.

As just mentioned, the advantage of closed filters is that they

lend themselves to filtration under pressure. Consequently the filters are, as a matter of course, constantly full, an even flow is assured, without any risk of overflowing, so that all that has to be done is to regulate the tap on the beak of the swan's neck so as to get the rate of flow desired, whilst in open filters both discharge and arrival taps must be adjusted simultaneously. Moreover, should the percolation through a single filter not completely purify the liquid, it may be run from the first filter to a second, and, if need be, even from the second to a third, thus doubling or even trebling the height of black through which the juice or syrup percolates.

In a well-conducted factory special filters should be allocated for the juices, whilst at the same time others are solely used for syrup. Indeed it has been ascertained that char does not behave in quite the same way with dense liquids as it does with light. It acts more efficiently on the former than on the latter. At one time manufacturers got into a deplorable habit as regards the purification of the juice. When a filter had been used for syrup they ran on juice on to the top of the syrup to force the syrup out of the filter, and that same filter was still used to filter juice as long as it came through clear. The objection to this method was that it redissolved in the juice a portion of the impurities left by the syrup owing to its great density. Analysis shows this very emphatically. Formerly sugar-beet factories were very rare. It is not therefore astonishing that these pernicious habits were introduced into the business. At the present day bone black has almost disappeared from beet-sugar factories, and in those exceptional cases where it is still used these defective processes have been amended by introduction of chemical control.

As soon as the juice begins to come through the filter turbid the process is stopped, and the contents of the filter washed with water until it does not mark more than 1° B. As the wash-water is very impure, only the first portion is sent to be treated, the remainder being used for pulping or diffusion, for washing the skimmings or the lime.

Loss of sugar in the bone black and its wash-water.—This is easily determined. The weight of bone black used and the number and capacity of the filters emptied during a given period are easily ascertained. The wash-water is about equal to half the bone black used, and the sugar left in the black is about equal to the sugar in the wash-water. All the data are therefore to hand for estimating the loss in question. It is, however, advisable from time to time to test the bone black after addition of carbonate of soda. The exhausted black is collected, dried, and weighed, so as to determine the ratio between the wet and the dry bone black.

XXXVIII.—TABLE GIVING THE PERCENTAGE OF LIME IN ANIMAL CHARCOAL, WORKING ON THE NORMAL WEIGHT OF 1.7 GRAMMES.

FOR USE IN THE SPECIAL TESTING OF ANIMAL CHARCOAL (SCHEIBLER).

Volume of Gas given off.	Carbonate of Lime in 100 Parts of Animal Charcoal, the Carbonic Acid being measured at the Temperature of—																		
	12° C.	13° C.	14° C.	15° C.	16° C.	17° C.	18° C.	19° C.	20° C.	21° C.	22° C.	23° C.	24° C.	25° C.	26° C.	27° C.	28° C.	29° C.	30° C.
1	0.80	0.79	0.79	0.79	0.79	0.78	0.78	0.78	0.77	0.77	0.76	0.76	0.76	0.75	0.75	0.74	0.74	0.73	0.73
2	1.88	1.87	1.86	1.86	1.85	1.84	1.83	1.82	1.81	1.80	1.79	1.79	1.78	1.77	1.76	1.75	1.74	1.73	1.72
3	2.96	2.92	2.91	2.90	2.89	2.89	2.87	2.86	2.85	2.83	2.82	2.80	2.79	2.77	2.76	2.74	2.73	2.72	2.71
4	4.01	3.94	3.98	3.96	3.94	3.92	3.91	3.89	3.87	3.85	3.83	3.81	3.79	3.77	3.75	3.73	3.71	3.70	3.68
5	5.07	5.05	5.03	5.00	4.98	4.96	4.93	4.91	4.89	4.86	4.84	4.81	4.79	4.76	4.74	4.71	4.69	4.67	4.65
6	6.11	6.09	6.06	6.03	6.00	5.98	5.95	5.92	5.89	5.86	5.83	5.81	5.78	5.75	5.71	5.68	5.65	5.63	5.61
7	7.14	7.12	7.09	7.06	7.00	6.99	6.96	6.92	6.89	6.86	6.82	6.79	6.75	6.72	6.68	6.65	6.61	6.58	6.56
8	8.17	8.14	8.11	8.07	8.03	8.00	7.96	7.92	7.88	7.84	7.80	7.76	7.74	7.68	7.64	7.60	7.56	7.53	7.50
9	9.19	9.16	9.12	9.07	9.03	8.99	8.95	8.90	8.86	8.82	8.77	8.73	8.68	8.64	8.59	8.55	8.50	8.46	8.42
10	10.20	10.16	10.12	10.07	10.02	9.98	9.96	9.88	9.83	9.79	9.73	9.68	9.63	9.58	9.53	9.48	9.43	9.39	9.34
11	11.20	11.15	11.10	11.05	11.00	10.95	10.89	10.84	10.79	10.74	10.68	10.63	10.57	10.52	10.46	10.41	10.35	10.30	10.25
12	12.20	12.15	12.09	12.03	11.98	11.92	11.87	11.81	11.75	11.69	11.64	11.58	11.52	11.46	11.40	11.33	11.27	11.22	11.16
13	13.20	13.14	13.08	13.02	12.96	12.90	12.84	12.78	12.72	12.65	12.59	12.53	12.46	12.40	12.33	12.26	12.20	12.14	12.07
14	14.20	14.14	14.07	14.01	13.94	13.88	13.81	13.75	13.68	13.61	13.54	13.48	13.41	13.34	13.26	13.19	13.12	13.05	12.99
15	15.20	15.13	15.06	14.99	14.92	14.85	14.78	14.71	14.61	14.57	14.50	14.42	14.35	14.27	14.20	14.12	14.04	13.97	13.90
16	16.20	16.13	16.06	15.98	15.91	15.83	15.76	15.68	15.57	15.53	15.45	15.37	15.29	15.21	15.13	15.05	14.97	14.89	14.81
17	17.20	17.12	17.04	16.97	16.89	16.81	16.73	16.65	16.51	16.49	16.41	16.32	16.24	16.15	16.07	15.98	15.89	15.81	15.73
18	18.20	18.12	18.03	17.95	17.87	17.79	17.70	17.62	17.53	17.45	17.36	17.27	17.18	17.09	17.00	16.91	16.82	16.73	16.63
19	19.20	19.11	19.03	18.94	18.85	18.76	18.67	18.59	18.50	18.40	18.31	18.22	18.13	18.03	17.94	17.84	17.74	17.61	17.55
20	20.20	20.11	20.02	19.93	19.83	19.74	19.65	19.55	19.46	19.36	19.27	19.17	19.07	18.97	18.87	18.77	18.66	18.56	18.46
21	21.20	21.10	21.01	20.91	20.81	20.72	20.62	20.52	20.42	20.32	20.22	20.12	20.01	19.91	19.80	19.70	19.59	19.48	19.37
22	22.20	22.09	22.00	21.90	21.80	21.70	21.59	21.49	21.39	21.28	21.17	21.07	20.96	20.85	20.74	20.63	20.51	20.40	20.78
23	23.20	23.09	23.00	23.00	23.00	23.00	23.00	23.00	23.00	23.00	23.00	23.00	23.00	23.00	23.00	23.00	23.00	23.00	23.00
24	24.20	24.09	23.98	23.87	23.70	23.65	23.54	23.48	23.31	23.20	23.08	22.97	22.85	22.73	22.61	22.48	22.36	22.23	22.12
25	25.20	25.08	24.97	24.86	24.74	24.63	24.51	24.39	24.26	24.16	24.04	23.91	23.79	23.67	23.54	23.41	23.28	23.15	23.02

Washing and revivification of bone char.—Bone black is revived in French sugar-beet factories by a chemical process followed by calcination in closed vessels.

The char after use contains a large amount of carbonate of lime, organic salts of lime, nitrogenous and non-nitrogenous matters,—in fact, soluble and insoluble salts.

The char, as it comes from the filters, is run into capacious wooden or masonry vats, and covered with water. Hydrochloric acid is then added. The acid dissolves the lime and the organic acids, decomposes certain salts, and restores the porosity of which the black was deprived by the substances which it had absorbed.

In certain countries, such as Russia, instead of using costly acid, a little more sugar is allowed to remain in the char, so that after a little time the contents of the vats ferment.

The carbonic acid which is evolved attacks the carbonate of lime, converts it into soluble bicarbonate of lime, and acts on the organic salts of lime in the same way. Moreover, as soon as the alcoholic fermentation ceases the lactic reaction sets in, and thus the chemical purification of the bone char is accomplished. The smell from these fermenting vats is horribly bad, consequently where hydrochloric acid can be got at a reasonable price it is much to be preferred.

The bone char is taken out of the vats and washed with a bulky volume of water in a washer, of which there are several varieties, which sometimes differ perceptibly from one another.

These washers were on the principle of a rudimentary Archimedean screw turning in a slightly inclined trough, and causing the black to ascend from below upwards, so as to meet a current of water flowing from above downwards.

Another widely-used washer consists of a horizontal cylinder bearing obstacles in its interior and turning on its axis. The black is rolled in the cylinder in the presence of a current of water flowing in an opposite direction, and issues washed at the opposite end of the machine to where it entered. This is a better system than the preceding, as the black is not crushed so much. There are several makers of machines on this pattern, which is much used both in France and Germany and other foreign countries.

Again, a very ingenious washer consists of a series of rakes in the shape of a hand which raises the black from stage to stage in a sort of inclined trough, divided by partitions into a series of consecutive compartments.

Whatever system of bone-char washing be adopted, in addition to char of the normal grist it yields more or less of fine char—char dust which is separated by the water and sold as manure.

The washed char is then treated in cylinders by steam from the boilers for a few minutes, which still further cleanses it. It is then

transferred to the revivifying furnaces, the rôle of which is to burn off in closed vessels the organic matter which it contains.

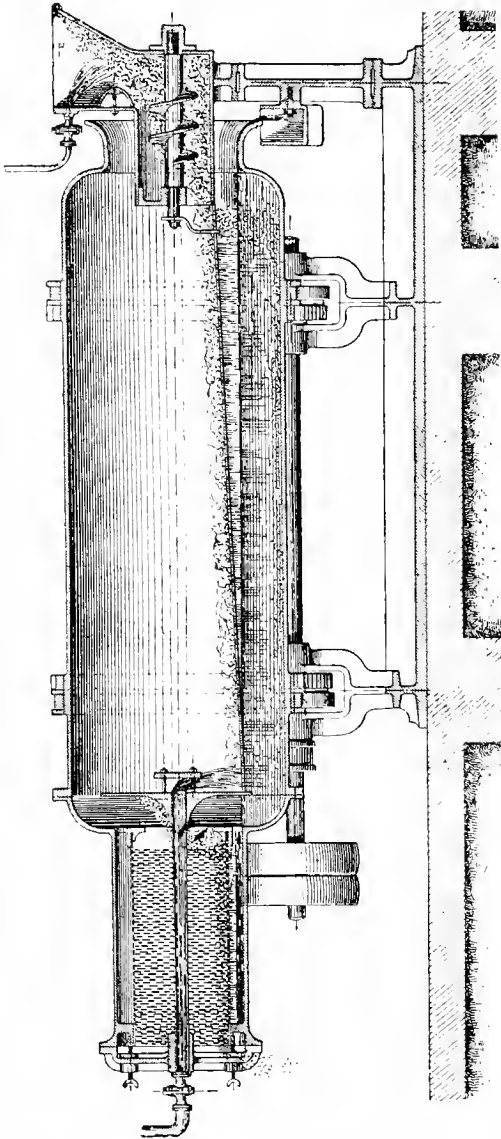


FIG. 42.—Bone-char washer.

The bone-black revivifying furnaces.—There are a great number of different forms of bone-char revivifying furnaces in existence. They all consist essentially of inclined or vertical cast-iron retorts, arranged in two rows in a furnace. The head of the retort comes out of the top of the furnace and rests on the surface of the masonry, which is covered with a large cast-iron plate, with as many openings as there are retorts. The lower end is closed by a movable, sliding, or hinged door.

The moist char is thrown on the cast-iron plate covering the top

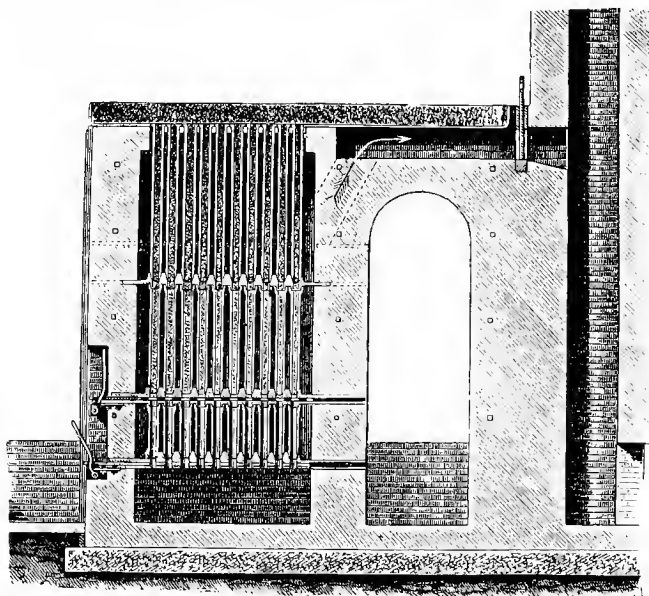


FIG. 43.—Animal-charcoal revivifying furnace.

of the furnace. The latter being heated to such a temperature that the retorts passing through it are heated to a cherry red, the water is slowly evaporated. The dry char is now shovelled into the retorts, and when sufficiently revivified the retorts are discharged into special barrows, in which it is transferred to the charcoal warehouse, to be used over again as required. So that the reburning may be carried on systematically, the retorts are partially emptied at regular intervals by withdrawing small consecutive portions by the bottom door, and they are filled from the top in proportion as the charge is withdrawn from underneath.

Revivified bone black denser and more compact than new bone black.—Revivified bone black is a little more compact than when new, because the temperature to which it has been brought has caused certain portions, more sensitive to the action of heat than others, to fuse.

The char cannot be revivified ad infinitum.—It thus happens that after being revivified a certain number of times the charcoal no longer exercises the decolorising properties for which it is esteemed. It is of no further use, being non-porous. It is spent. This spent char is of no further use except for the manufacture of artificial manure, for which the high percentage of phosphoric acid pre-eminently fits it, but it makes a very dirty sooty-looking manure, to which farmers, with their inveterate prejudices, do not take to kindly. Spent char is also sold to paint manufacturers, who “fake it up” by striking a small percentage of Prussian blue on to it; or incorporate a small percentage of ultramarine blue, when it is ground. Owing to its being much harder than when new, for reasons just explained, it is much more difficult and more costly to grind than new black. Nevertheless, some paint manufacturers still grind the spent char with water in the manner described, pass it sometimes through a perforated plate, in the form of drops, and sell it as “*Superior Genuine Ivory Black.*” Spent char is not fit for use in making boot blacking; indeed, for paint purposes it would be more economical in the end to use a superior new bone black, which, if dearer at the outset, would entail far less labour and yield a far superior product. It is of little use for a paint manufacturer to try to match a paint made from new char with one made from spent char. No amount of faking will make but a very bad imitation.

Bone black superseded by mechanical filters.—Such is briefly the history of animal-charcoal filtration in sugar manufactories. The methods generally yielded but little result from a purification point of view, because enough of it was not used. To such an extent was this the case that certain factories, seeing the small effect it produced, no longer use it except as a mechanical filtration agent to arrest the suspended impurities. It was then that the mechanical filters, properly so called, appeared on the scene and displaced animal charcoal.

Mechanical filtration.—As has just been described, mechanical filtration consisted in the beginning in the use of a small quantity of animal charcoal, and generally of old charcoal, having nothing but its colour to indicate its origin, and equally devoid of both porosity and bleaching effect.

Puvrez bag filters.—Attempts were therefore made to replace char by coarse sand. Gravel did not succeed very well, and experiments with it did not last long, for Puvrez bag filters came on the scene in 1884-85.

It has been contended that the Puvrez bag filters were only imitations of the old Taylor's bag filters. But, in fact, it must be borne in mind that Taylor's filters (p. 339) never gave good results when tried in sugar-beet factories. On the other hand, Puvrez, by causing the dull turbid juices to flow through the pores of the bag-shaped filter cloth *without any exterior sheath* and under a feeble pressure, barely three to five feet of liquid, was able, with that simple method of filtration, to impart to these juices a limpidity unknown up to that time. Therein consisted the novelty, which made a great commotion. Puvrez's secret consisted in the careful selection of the filter cloth, a cross fabric specially designed for this purpose and answering perfectly all the requirements of a good filter cloth.

Puvrez's bag filters consisted of simple cloth pipes from 6 to 13 feet long, closed at one end and joined at the other to the shank of a tap. The bag was laid in a trough and rested on an iron grating, which kept it from touching the bottom of the trough. A series of pockets were arranged side by side, each in a similar compartment, and received the juice of the filter presses collected in a shallow tank. The pockets, filled with juice, formed a big pipe, which exuded a perfectly clear liquid from the whole of its surface. But it is needless to state that such a cloth will not filter muddy liquids, because the pores are quickly closed up. The object of such a filter is to clarify liquids which are no more than cloudy, consequently containing but very little suspended matter.

Consequently, when the so-called limpid juices of the sugar factory are run into the pockets they leave a certain amount of deposit, invisible in the bulk of liquid, but very tangible when collected. Moreover, juices filtered in this way give a much superior product to those which have not been so filtered, and quite equal to the filters with an insufficient quantity of bone black, so long used in sugar factories.

Puvrez bag filters, therefore, opened up quite a new field to manufacturers, and filtration through cloth, much simpler and more economical than filtration through bone black, immediately became general.

Puvrez bag filters were clumsy and inconveniently arranged. Their renewal was a dirty job, because, when the juices failed to pass through, it was necessary to empty them of all their contents by undoing the string which held them to the union of the tap, and this was unpleasant for the workmen, who burned their hands and threw mud in every direction. Attempts were therefore made to remedy these defects by imparting a more mechanical mounting to the bags.

Daneck's mechanical filter.—The first mechanical filters fulfilling all the necessary requirements were constructed about 1887 by the firm of Breitfeld Daneck of Prague. They were invented by an engineer of that firm residing in Russia, M. Prokché, and are known as Daneck filters.

The filter consists essentially of a square wrought-iron tank 3' 3" x 3' 3" x 3' 3". The bottom consists of two surfaces inclined at 45°, and the top is closed by a hinged cast-iron door with counterpoise, making a tight joint by means of an indiarubber washer, and tightening screws arranged all round about it. The whole rests on four feet.

The pockets, which consisted of cloth filter bags hanging on a corrugated sheet of iron, were arranged inside the tank. The sheets of iron with their bags were suspended, to the number of about 40, in the tanks by means of bars resting at each end on special supports. The bars were iron pipes like gas-piping, and they were covered by the upper edge of the bag, which was folded round and fixed to them. The sheet iron penetrated into these tubes, leaving interstices which put the interior of the tubes in communication with the interior of the bags. Finally, one end of the tubular bar was closed, and the other open. In the top of the tank there were arranged, in two horizontal lines very close to each other, as many tubulures as there were bags suspended in the interior. The tubular bars penetrated into these tubulures by their open extremity by means of an indiarubber connection, whilst the closed extremity rested on a screw support, which fixed it firmly in its place by pressing against the tubulure. Underneath the tubulures on the outside of the tank was a trough.

Its mode of action was as follows. The juice to be filtered is run into the closed tank, under a pressure of 6 to 10 feet of liquid. The juice passed from the exterior to the interior of the cloth bags, entered the tubular bars, traversed the tubulure, and issued from the tank by running into the trough.

Consequently the deposit was made on the outside of the cloths and not in the interior, as in Puvrez bag filters. Moreover, as the bags could be packed very closely, a large filtering surface was combined in a small space. Again, as the tank was closed no overflow could be feared, the filtered juice being only cooled very slightly; no steam is formed on the outside, and all that had to be done as regards cleaning was to close the juice tap for running in the juice, empty the tank, open the lid, remove the corrugated sheets of iron with their bags and replace them by others. All this was effected rapidly and neatly, and presented every guarantee for the subsequent effectual cleaning of the bags.

Daneck's filter may therefore be said to have attained perfection from the very first. All that has been accomplished since is only more or less successful imitation. One of these, however, Phillippe's filter, shows some slight improvement in the mounting.

In Phillippe's filter (Fig. 44) the lid is dispensed with, as well as its cumbersome counterpoise. The top of the tank is closed, and is pierced by a series of holes to afford passage to the bags and their

wavulating iron sheet. The hollow bars do not enter into the tank, being longer than the holes. A hood is then placed over these, which covers them completely, and which bears on the front of it a tubulure for the discharge of the juice. The joint of the cap is made on the

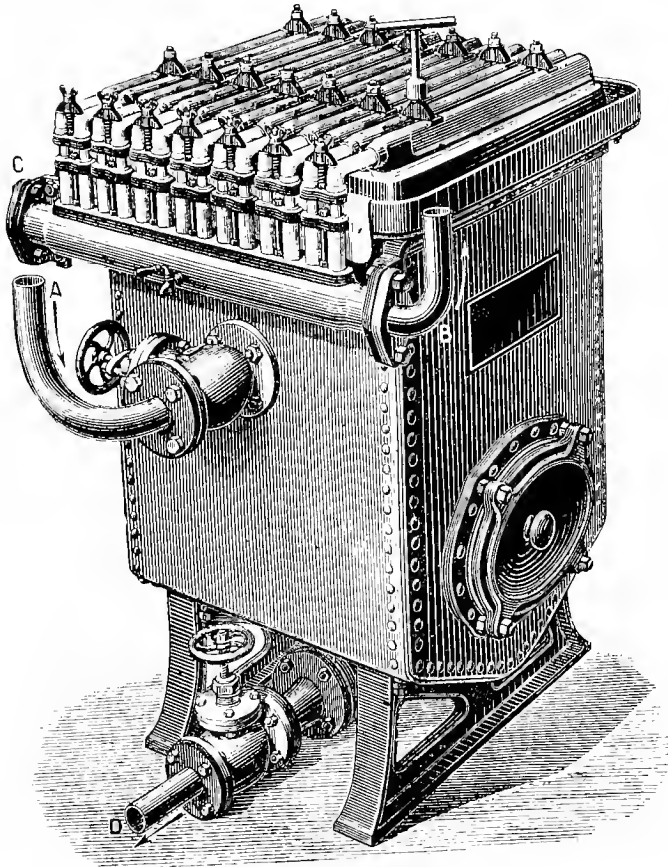


FIG. 44.—Mechanical filter (PHILIPPE).

top of the tank by means of the cloth itself, which is specially arranged for the purpose, and by means of pressure screws easily undone. The renewal of cloths only requires the lifting of the caps; and if, for example, one of the cloths is pierced, it may be removed without interfering with the joints of the others. Consequently

Phillippe's filter has some advantages over Daneck's in the handling of the framework.

Another filter, namely Kazelowski's, differs in no way from Daneck's filter, except that the corrugated wrought iron is replaced by corrugated wire gauze. Certain engineering firms nevertheless make this filter only, but its yield is just the same.

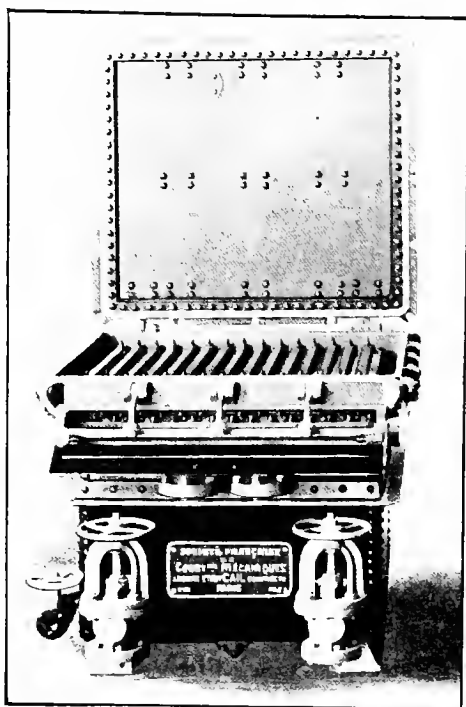


FIG. 45A.—Kazelowski's filter ('MAIL).

Generally each frame of each of these filters represents a square metre of filtering surface. Filters, therefore, with twenty or forty frames represent as many square metres of surface.

Mechanical filtration has been applied to the juice of the first carbonatation, to that of the second, as well as to the syrups. As, owing to its alkalinity, first carbonatation juice filters with difficulty, and requires a great extent of filtering surface, this filtration is often suppressed, but wrongly so. The majority of factories have no filtra-

tion plant, except for concentrated juice before evaporation and for syrups.

Summary.—Mechanical filtration is a very simple and comparatively compact portion of the work, and is one of the most interesting in the purification of the juice. When well performed, it largely increases the yield of the beetroot in that grade of sugar known as “firsts.”

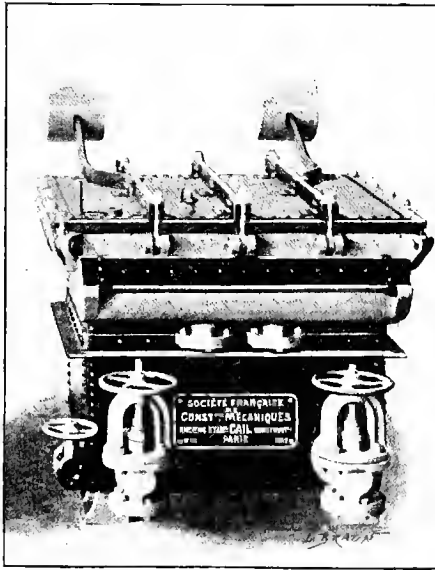


FIG. 45B.—Kazelowski's filter (C.A.L.).

The accompanying table shows the progress made in France in filtration methods. The table speaks for itself and needs no comment, further than that the use of bone-black filters would appear to be again being resorted to by manufacturers since the season 1897-98, and, what is peculiar, more especially so in the case of open filters.

[TABLE XXXIX.]

XXXIX.—PLANT USED IN FILTRATION OF BEET JUICE IN FRANCE,
1884-1900.

Season.	Number of Filter Presses.	Number of Bone Black Filters.		Number of Filter Frames and Filter Bags.
		Open.	Closed.	
1884-1885	2111	2061	583	...
1885-1886	2117	1666	630	815
1886-1887	2106	1301	556	1966
1887-1888	2016	840	448	1626
1888-1889	2055	729	391	1812
1889-1890	2102	553	364	1791
1890-1891	2078	369	336	1811
1891-1892	2147	249	290	1910
1892-1893	2203	176	233	1951
1893-1894	2194	145	156	1998
1894-1895	2266	98	142	1982
1895-1896	2247	79	125	1993
1896-1897	2319	87	160	2106
1897-1898	2311	217	169	2035
1898-1899	2356	204	161	2169
1899-1900	3398	238	140	2215

CHAPTER VII

THE CONCENTRATION OF THE BEET JUICE TO SYRUP IN THE MULTIPLE-EFFECT EVAPORATION VESSELS (IN VACUO)

CONCENTRATION OF THE JUICE BY EVAPORATION IN VACUO—THE VACUUM PAN—MULTIPLE-EFFECT SYSTEMS OF EVAPORATION IN VACUO

DIFFERENTIATION of evaporation from distillation.—The general object of evaporation is to increase the original strength of a liquid in solids from r_y per cent. to a greater strength r_n by elimination of the water. In *evaporation* it is sought to recover the fixed residue; in *distillation*, the recovery of the volatile ingredient is the main object, that of the fixed residue a secondary one. The amount of water to be evaporated, in any given instance, may be ascertained as follows:—

If there be r_y kilogrammes of solids in 100 kilogrammes of liquid, and if the r_y kilogrammes are to become r_n per cent. in the concentrated liquid, then the *weight U of the concentrated liquid* is given by—

$$r_y : U = r_n : 100, \text{ or } U = \frac{r_y 100}{r_n}$$

Again, the *weight of water to be evaporated* from 100 kilogrammes of liquid is

$$100 - U = 100 - \frac{r_y 100}{r_n} = 100 \left(1 - \frac{r_y}{r_n} \right)$$

and the weight of water to be evaporated from W kilogrammes of a liquid which contains r_y per cent. of solids, in order to concentrate it to the strength of r_n per cent., is

$$W - U = W \left(1 - \frac{r_y}{r_n} \right)$$

Example.—1000 kilogrammes of juice containing 10 per cent. of solid matter is to be evaporated to such an extent that the residue will contain 80 per cent. Then—

$$W - U = 1000 \left(1 - \frac{10}{80} \right) = 875$$

The accompanying table shows the quantity of water in pounds or kilogrammes to be evaporated from 100 lbs. or kilogrammes of juice

or weak saccharine liquors containing from 1 to 20 per cent. of solid matter to so concentrate them as to bring the total solids to 80 per cent.

XL.

Strength of Juice or Saccharine Liquor in Solids per cent.	Kilos. or lbs. of water to be evaporated per 100 kilos. or lbs. of juice to bring the total solids to 80 per cent.	Strength of Juice or Saccharine Liquor in Solids per cent.	Kilos. or lbs. of water to be evaporated per 100 kilos. or lbs. of juice to bring the total solids to 80 per cent.
1	98·75	11	86·25
2	97·50	12	85·00
3	96·25	13	83·75
4	95·00	14	82·50
5	93·75	15	81·25
6	92·50	16	80·00
7	91·25	17	78·75
8	90·00	18	77·50
9	88·75	19	76·25
10	87·50	20	75·00

It may at first sight be rather surprising to those who have not studied the matter, that, after concentrating a liquid which already contains 20 per cent. of solid matter by vaporising three-fourths of its original weight, it should even then only contain 80 per cent. of solid matter. But a little reflection will soon show the truth of the statement.

The outlay on fuel a preponderating item of expenditure.—From the foregoing statement of the amount of water to be evaporated to attain even such a comparatively low degree of concentration as 80 per cent. it will be readily seen that the coal bill undoubtedly forms, even at the present day, one of the largest items of the expenditure incidental to the working, carrying on, and maintenance of a beet-sugar factory. The reason for this is not far to seek. It must be borne in mind that, in addition to the large amount of steam used up by the very heavy motor-power required to drive the machinery, and by the continued reheating of the juice over and over again, both of which operations use up and consume much steam, there is still another and final operation which of necessity also involves a great consumption of steam and consequently of fuel. It is, in fact, in addition to the above onerous operations, absolutely necessary to completely evaporate almost the whole of the water in which the sugar is dissolved, in order to get a syrup with a tendency to

crystallise. From a historical point of view, the history of the evaporation of the sugar juice may be divided into three periods—

1. The period of the open pan over a naked fire.
2. The period of the Howard or single-effect vacuum pan.
3. The multiple-effect system of the present day.

1. *The old-fashioned costly wasteful system of evaporation in open pans over a naked fire.*—The boiling down of the juice to the crystallisation point was for a very long period, in the history of the sugar industry, carried on, with free access of air, in open pans over a naked fire. This rudimentary process has given rise to the remark that the beet-sugar factories of those days were nothing more nor less than large confectionery works, where everything was carried on in as simple a style as possible, but, all the same, not without leaving a considerable amount of sugar in all the residues. Needless to say, the process therefore entailed a great loss of sugar. But this was not all. There was great waste of fuel in addition to the loss of sugar. In fact, it involved the combustion of 400 lbs. of coal to obtain the paltry amount of 4 to 5 per cent. of sugar (40 to 50 lbs.) from 1000 lbs. of beets. It will not, therefore, be a matter for surprise that sugar was quite beyond the reach of the poor in those countries whose supplies of sugar depended on beet sugar produced in this way. The utilisation of flue gases for heating purposes is discussed by Hansbrand, *Evaporating*, etc., pp. 12–17, where certain comparative data are given as to the working value of different fuels; and on pp. 39–55 much valuable information is given regarding heating by steam coils and steam jackets.

1828. *First use of steam in beet-sugar factories (for heating and evaporating purposes).*—A goodly portion of the nineteenth century had passed away before steam was first utilised in beet-sugar factories, but this utilisation was even then only a very partial one. It will no doubt nowadays be a matter for surprise that the first use to which the steam was put was *not to drive the machinery, but to heat the steam jackets and steam coils*, which had come into vogue for heating the defecation pans. But the industry was then only in its infancy. There were only fifty-eight factories at work in France, and thirty-one being built, whilst the area under beets was only between 9000 and 10,000 acres.

The introduction of steam coils paves the way.—It was about this time that *Moulfarine* and *Pecquer* (p. 187), two Paris engineers, quite independently of each other, made successful efforts to evaporate the juice by means of steam coils circulating in the liquid in the bottom of flat pans, which suit this kind of work. *Moulfarine* and *Pecquer's* innovation constituted the starting-point from which afterwards sprung the use of steam engines in the beet-sugar factories of France.

The introduction of the steam engine retarded by ignorant unfounded fears of explosion.—But the day of the steam engine in beet-

sugar factories, however, was not yet. Fear of explosions and hazy ideas as to their causes for a long time retarded its introduction, and the French people were so much imbued with preconceived prejudices against it that manufacturers as a class did not want to have anything to do with it, because *steam engines were liable to explode*. It was not at that time so generally known and understood that what causes an explosion, when one does occur, in connection with a steam engine, is not the steam engine itself, but the boiler. But they already had and were actually using the boiler, the really and only dangerous element of the combination; but they did not want the engine, the comparatively harmless appliance—at least so far as explosion is concerned.

Howard's vacuum pan could not at first be wrought in France for want of engine to work the pump.—The retarding influence of this ignorant prejudice will be better appreciated when it is mentioned that Howard's vacuum pan, which had been in use in Britain since about the year 1800, could not be introduced into the French factories for want of an engine to work the pump. In Howard's pan the evaporation was effected in a vacuum, thus avoiding the enormous destruction of sugar incidental to a boiling down of the juice over a naked fire. *Howard's vacuum pan* therefore gave a large yield of sugar, and not only so, but sugar of a superior quality; but they could not be used in France because their use entailed the simultaneous employment of a steam engine to produce the vacuum, and that is the reason why up to 1830 or even 1840 there were no vacuum pans in France.

Derosné's and Degrande's modification of Howard's vacuum pans lowers the consumption of fuel $37\frac{1}{2}$ per cent.—Howard's pan, as originally constructed, will be described in that section of this treatise which deals with *sugar refining*, but, as adopted in France, they were provided with a Derosné condenser, and were termed Derosné's machines. Their adoption in France was attended by such a satisfactory result that the consumption of coal in beet-sugar factories went down from 400 lbs. to 250 lbs. per 1000 lbs. of beets—a saving at the very outset of $37\frac{1}{2}$ per cent. on the costly item of fuel alone.

1852. *Introduction and adoption of the multiple-effect vacuum evaporation principle effects a further reduction of 60 per cent.*—Multiple-effect vacuum plant was first introduced into the beet-sugar industry in 1852, and it is this identical plant which, improved by its inventor in 1882, is still in general use at the present time. Rillieux in this way lowered the consumption of coal to less than 100 lbs. per 1000 lbs. of beets. In other words, he reduced the amount of fuel consumed by 60 per cent.

Summary.—The above brief epitome of the history of evaporation in beet-sugar factories is necessarily also that of the introduction of

machinery and plant on the large scale as it exists at the present day, and which has converted them into engineering establishments of the first rank, as will be seen from the Table XLIV., etc. But, coming back again to the technical aspect of evaporation, it will be necessary to follow it from the beginning to the present time.

The useful effect of the heat of combustion of fuel.—In a good boiler 1 kilogramme of coal produces about 8 kilogrammes of steam. To evaporate 8 kilogrammes of water, about 5000 calories must be developed. Coal, in burning, produces about 7500 calories. There is thus a loss of 2500 calories, or a third of the heat given off by the coals. These 2500 calories are absorbed (1) by the cold air entering beneath the grate in greater quantity than is required by theory, and escaping into the chimney in the form of hot gases; (2) by radiation; (3) by any cause which interferes with the good working of the fires—bad stoking, boiler encrustation, bad draught, etc.

The great loss of heat entailed in heating swing or tip-tilting pans over naked fires.—The loss of heat is far greater beneath a pan, it being impossible in the case of this class of vessel to apply all the improvements in the construction of a furnace that can be done with a boiler; and as sugar pans were on the swing or tip-tilting principle, this was more difficult still, as they could not be built into the masonry. The bottom of the pan therefore was alone heated.

The wasteful state of affairs in 1825.—Now, as 1000 kilogrammes of beet yield about 800 kilogrammes of juice, say 700 kilogrammes of water to be evaporated after reheating, it would be necessary (1) to burn about 40 kilogrammes of coal to defecate the juice, and (2) 280 kilogrammes to evaporate it, say 320 kilogrammes of coal for the complete treatment of the juice, an amount which very soon rose to 400 kilogrammes, the more so when extreme care was not taken in heating. This was the actual state of affairs in the régime of the old sugar factories in 1825.

Improvement effected by introduction of steam.—Matters improved, however, as soon as steam was introduced into factories; the production of the necessary heat was effected more economically, since water can be converted into steam under much better conditions in a steam boiler, and this steam brings its evaporative energy to bear in the steam coils of the evaporation and defecation pans with a much less perceptible loss of calories than naked fire.

The amount of useful heating effect of the old steam boilers.—The steam boilers of those days barely evaporated 7 lbs. of steam per lb. of coal. To heat 800 lbs. of juice to boiling-point it was necessary to condense 140 lbs. of steam, requiring 20 lbs. of coal. To evaporate 700 lbs. of water it requires 700 lbs. of steam, say 100 lbs. of coal. The engines consume an amount of steam equal to about 40 lbs. of coal, say, in all, about 160 lbs. of coal. Radiation and considerable loss in working had also to be allowed for, because all the vessels were of

copper as brilliant as it was possible to make it, so that it might look well, for they did not take into account what this polishing cost; and we thus arrive at the total, including everything, of 250 lbs. of coal, of which 50 lbs. at least could be economised. But at that time they did not go so fully into such matters. The accompanying tables, embodying the results of recent experiments, will be read with interest in this connection.

XLI.—BOILING-POINTS OF PURE AND IMPURE SOLUTIONS OF SUGAR.

Degrees Balling.*	Boiling-points at 760 Millimetres Pressure.		
	I. Solutions of Pure Sugar.	II. Juice and Syrup.	III. Low Products.
	° C.	° C.	° C.
10	100·1	100·2	100·3
20	100·3	100·4	100·6
30	100·6	100·8	101·1
40	101·1	101·4	101·7
45	101·4	101·8	102·2
50	101·4	102·2	102·7
55	101·9	102·8	103·4
60	102·4	103·5	104·2
65	103·1	104·4	105·3
70	105·3	105·8	106·8
75	107·4	...	108·5
80	110·3
85	114·5

* For corresponding densities, see Table I. p. 3.

About 1830 Rillieux began to study the evaporation problem, which he solved by his multiple-effect system *in vacuo*.

An American by birth, but the discovery made in France.—Rillieux, it appears, was born in America, but of French origin, and all his experiments were carried on in France. As early as 1830, although he was then but quite a young man, he was the best steam-engine engineer in Paris, and had already taken out patents for inventions.

Neither French nor American capitalists would assist.—However, Rillieux was unable to get his invention taken up in France. In despair he tried America, but with no more success at first.

His plant at last tried on an American homestead.—In fact, it was not until about fifteen years later, namely about 1845, that he was able to find an American manufacturer who was willing to try his apparatus on his homestead. From that time forward a number of double- and triple-effect apparatus were constructed in America.

XLII.

	1892.			1893.			1896.
	I.	II.	III.	IV.	V.	VI.	VII.
Results of Vaporisation of Water Experiments (Schmidt).							
Effective pressure (in kilogrammes)	4.59	4.74	5.36	5.20	4.23	4.23	4.37
Temperature of the water (in degrees Centigrade)	95	85.8	95	92.7	86.3	86.3	81.1
Ratio—Heated surface— Surface of grate	39.5	52.3	53.5	45.1	79	44	73
Combustion per hour and per square metre of surface of heated surface (in kilogrammes)	1.70	1.38	1.58	1.67	1.17	1.71	1.71
Combustion per hour and per square metre of surface of grate (in kilogrammes)	67.4	72	84	75.5	93	74.7	126
Furnace residuum—Actual ash	13.5	9.7	10.0	12.8	10.7	10.6	9.9
Ash of the coal by analysis per cent.	5.6	5.4	7.6	5.7	7.0	7.0	7.9
Steam produced per hour and per square metre of heated surface (in kilogrammes)	15.9	13	14.5	15.4	11.1	15.3	15.9
Steam produced per hour and per square metre of plane of water (in kilogrammes)	288	205	...	268	232	261	328
Steam produced per hour and per cubic metre of steam chamber (in kilogrammes)	601	376	...	612	543	524	779
Apparent yield of steam per kilogramme of coal.	9.3	9.4	9.2	9.2	9.5	8.97	9.3
100.0 { Utilisation per cent.	64.3	64.2	64.5	64	69.2	65.3	67.3
Loss in chimney	24.4	29.3	29.4	...	23.9	27.9	17.3
Other losses per cent.	11.2	6.5	6.1	...	6.9	6.8	15.4
Price of coal per ton delivered at the factory (in francs)	21.0	20.7	29.5	21.25	20.16	20.16	21.45
Price per 1000 kilogrammes (metric ton) of steam (in francs)	2.24	2.19	3.20	2.31	2.12	2.25	2.31

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XLIV.

Period.	Evaporation of Juice.		Boiling down of Syrups.				Total Number		
	Number of Factories		Number of		Number of Factories		Number		
	Possessing Triple-effect.	Using Open Pans exclusively.	Triple, Quadruple, and Quintuple-effect Plants.	Open-air Pans.	Possessing Granulating Pans.	Using Open-air Pans only.	Number of Vacuum Pans unfit for making Granulated.	Sets of Plant for making Granulated.	Open-air Pans.
1884-85	401	48	507	237	372	69	17	445	437
1885-86	380	33	411	141	362	48	3	448	385
1886-87	367	24	367	115	357	32	2	437	342
1887-88	365	10	384	72	360	14	1	451	294
1888-89	372	8	399	53	367	12	1	470	293
1889-90	368	5	395	39	368	5	...	479	277
1890-91	376	1	409	3	375	2	...	494	244
1891-92	367	1	391	3	366	2	...	499	231
1892-93	367	1	393	3	367	1	...	517	220
1893-94	369	1	396	3	368	2	...	538	165
1894-95	366	1	394	3	366	1	...	556	149
1895-96	356	...	380	...	356	550	137
1896-97	413	529	...
1897-98	376	476	...
1898-99	383	505	...
1899-1900	364	540	...

Howard's vacuum pans (simple effect).—It is necessary to anticipate matters a little and to go ahead of the course of working in a sugar-beet factory, to speak, in the first place, of simple-effect vacuum pans, which are the pans used for boiling down the syrup, the boiling operation being that which immediately follows the evaporation of the juice. But the natural order of description of evaporating vessels *in vacuo* leads us to speak first of the former.

Moulfarine and Pecquer's flat pans heated by a steam coil.—It has been already mentioned (p. 179) that *Moulfarine* and *Pecquer*, two Paris engineers, each invented independently evaporation vessels consisting of flat pans heated by steam coils to evaporate the juice.

The evaporation problem in this instance is easily solved. To evaporate a liquid, it must first be brought to the boil in order to then supply it with the necessary calories to transform it into steam.

Definition of a calorie.—To heat 1 kilogramme of water 1° C. it is necessary to apply to it a certain amount of heat, which is taken as unity, and which is called a calorie.

To heat n kilogrammes of water t degrees, $n t$ calories of heat must be supplied.

At the atmospheric pressure, if it be desired to evaporate 1 kilogramme of water, the experiments of Regnault and other natural philosophers have shown that, when this water is at 100° C., it requires 537 calories.

Latent heat of steam.—To evaporate, therefore, n kilogrammes of water, it requires $n \times 537$ calories. This number 537 is known as the latent heat of steam, which varies with the temperature of the steam formed. When steam condenses, it gives up a certain number of calories. Regnault has given the formula—

$$L = 606.5 + 0.305 T$$

L being the total heat, and T the temperature of the steam.

When steam condenses it does not give up its total heat, but that total heat minus the calories contained in the condensed water. As at the moment of condensation the water formed is at the same pressure, consequently at the same temperature as the steam, that is to say, at T degrees, the number of calories abandoned by the condensed steam is—

$$L = (606.5 + 0.305 T) - T + 606.5 - 0.695 T.$$

Therefore, in evaporation with free access of air by means of steam, it is necessary to supply the water to be evaporated with $n(t + 537)$ calories, and to do that to condense in the coils x kilogrammes of steam yielding $x(606.5 - 0.695 T)$; we therefore get—

$$n(t + 537) = x(606.5 - 0.695 T).$$

Steam calculations.—The above formula is that which enables us to calculate the quantity of steam required to evaporate the water contained in the juice. Some other elements, such as the specific heat of the juice, enter into this formula, but we will not complicate

it further, our object being to make the rationale of the calculations understood.

The reasoning may be still further simplified:—1 kilogramme of water at 100° C., in being converted into steam, absorbs 537 calories; 1 kilogramme of steam at 100° C. in condensing abandons 537 calories. It is therefore necessary to condense 1 kilogramme of steam in order to evaporate 1 kilogramme of water. That is, however, not quite exact; nevertheless, as the difference is not very great, one can reason in that way without being very far out. Moreover, the preceding formula says exactly the same thing, because if we take water at 100° C. we have $t=0$; with the steam at 100° C. we have—

$$n(537) = x537.$$

$$\text{Hence } n = x.$$

In evaporation calculations it is necessary to take t and T into account at each moment, as they continually vary. But in the present case, it being given that the steam used in sugar factories in the time of the open pans was low-pressure steam, and that the juice was run into them hot, the above reasoning was quite sufficient.

Accordingly, in evaporating steam with free access of air, for every kilogramme of water evaporated it is necessary to condense 1 kilogramme of steam. For elaborate data relating to evaporation by means of steam coils and steam jackets, see Hansbrand, *Evaporating and Cooling*, pp. 39–55, to which the reader, and especially the sugar-works engineer, is specially referred.

The principle of Howard's vacuum pan.—Long before Pecquer's time, Howard (about 1800) constructed and used pans for boiling sugar *in vacuo* (*vacuum pans*): This apparatus will be described fully and its method explained under Sugar Refining, but for purposes of illustration and comparison it is necessary to state here that these vessels consisted of steam-jacketed pans covered by a dome, in which a vacuum was made. The heating steam entered the jacket or false bottom. The steam formed by the juice, which of late has been termed the juice steam, was led from the dome through a pipe to a condenser.

The vacuum was produced by the condensation of this steam in a condenser placed at the continuation of the exit pipe, and was kept up by an air-pump.

The principle of the condensation vacuum.—The vacuum question requires here a few explanations, which are generally applicable to all forms of condensers. It is demonstrated in physics that when a vessel in which steam is generated is perfectly closed, such as a glass flask in a laboratory or any kind of a boiler in a factory, if the steam filling the boiler be suddenly condensed a perfect vacuum is formed above the boiling liquid.

The effect of gases dissolved in liquid being evaporated on the condensation vacuum.—*Necessity for air-pump.*—Accordingly it would

only be necessary to condense the steam in a boiler to get a perfect vacuum therein. But in actual practice that is not altogether correct. Boiling liquids always contain dissolved gases or produce them, when these liquids, like beet juice, are of an organic nature. If, therefore, the steam be in some way or other condensed, the gases still exist, and if they be not removed the vacuum becomes less and less perfect. There is a gradual change from the space in which there is principally steam to that in which there is chiefly air through a space in which they are mixed. Hence the necessity to have a pump as a continuation of the condenser to remove the gases. Accordingly, the condensers must be provided with a pipe through which the air is drawn from their interior to the air-pump. This pipe must not be obstructed by water, since the variation in the pressure and amount of air or gas introduced into the condenser would cause currents backwards and forwards in this pipe so as to equalise the pressure. The presence of water in the pipe would, in fact, prevent the free extraction of the air by the pump, and thus cause irregular working.

Capacity of pump.—The capacity and function of air-pumps is dealt with at length by Hausbrand, *Evaporating*, etc., pp. 338–397. It is, however, necessary to give some few details at this point. This pump ought to have, in fact, a capacity proportional to the quantity of gas and air to be removed. Now, in the present instance, the condenser is a cylinder into which the steam enters, and into which a spray of cold water is injected. This cold water seizes hold of the steam, but in doing so it becomes hot, and this liberates all the air which it contains,—and it is known that water contains $\frac{1}{20}$ of its weight of air in solution. Consequently, when a cold-water injection condenser is used it is necessary to have a pump as a continuation of the condenser capable of removing not only (1) the gases coming from the juice itself, but also (2) the air dissolved in the condensing water. This pump, called an *air-pump*, ought to be of rather considerable proportions, the more so as the injection water represents 20 to 25 times the volume of steam to be condensed.

The relation between the capacity of the air-pump and the volume of the water injected.—Horsin-Deon has determined that the air-pump ought to develop per second a volume $12\frac{1}{2}$ times greater than the volume of the water injected. (Hausbrand, moreover (*Evaporating*, etc., pp. 344–376), gives elaborate information and data in regard to this important point.) This proportion corresponds to the most perfect working practically observed in the better class of condensers.

The component parts of Howard's evaporation system in vacuo.—Howard's vacuum pan therefore consists in principle of (1) a condensing surface, on the one side of which is the heating steam, and the other the juice to be evaporated; (2) a closed chamber in which is formed the steam from the boiling liquid; (3) an injection condenser, and (4) an air-pump.

The useful effect of such a system.—What will be the amount of steam required for evaporation under such conditions? It is evident, says Horsiu-Deon, that it will be perceptibly the same as for a pan with free access of air. It will, however, be a few degrees less, because the liquid in ebullition boils at a slightly lower temperature, but the difference is not very appreciable. He therefore concludes that Howard's vacuum pan is of very little interest *from a coal-saving point of view*. But, no doubt, this is due somewhat to prejudice. Further information as to the principles, advantages, and disadvantages of boiling in a single effect are given in Hausbrand's *Evaporating*, etc., pp. 56–61.

A higher-grade sugar and increased yield obtained by lowering the boiling-point.—The essential advantage obtained by distillation *in vacuo* is the better conditions under which the boiling syrup is placed and the better yield in sugar obtained from it. In the case of other organic products, *e.g.* milk, blood, sugar, and albumen, they can only be satisfactorily evaporated *in vacuo*. It is well known that when a vacuum exists above a liquid it boils at a lower temperature than under the atmospheric pressure, and the more perfect the vacuum the lower is the temperature in question.

Injurious effect of hot steam on organic liquids boiled down in contact with air.—Now it is an ascertained fact that the syrups formed by the concentration of organic juices are very easily decomposed by heat. Sugar syrups in particular are much affected under the action of heat,—the sugar is destroyed, it caramelises, and a beautiful straw-yellow syrup is quickly changed to a black under the action of very hot steam with free access of air.

Summary of beneficial results obtained by evaporation in vacuo.—One of the most favourable conditions conducive to good working, therefore, is to so lower the boiling-point of the syrup that the sugar is not in any way injured, since, by acting in this way, a far superior grade is obtained than when the syrup is boiled with free access of air. Moreover, a surplus yield is obtained consisting of all the sugar saved from destruction. That was the problem which Howard set himself to answer, and he solved it in a very fortunate manner with his vacuum pans.

The great safety from injury to the sugar in boiling at a lower pressure than that of the atmosphere.—As syrup boils *in vacuo* at a low temperature, it is therefore useless to heat it to the *high* temperature that was done in pans freely open to the atmosphere. All that has to be done is to run in steam, at a temperature a little above the boiling-point of the syrup, into the steam jacket in order to make it boil. That is, in fact, what is done in a well-constructed apparatus with sufficient heating surface. Moreover, if low-pressure steam—that is, low-temperature steam—be applied, there is much less chance of destroying the sugar, a condition highly conducive to an increased yield.

Peculiarities and progressive alterations in the construction of Howard's vacuum pans.—Howard's vacuum pans had an extensive enough heating surface to accomplish this, as they were very wide and contained but a very small quantity of syrup. They met the wants of the small factories of their time. Later on, however, in order to make the capacity of the vacuum pans keep pace with the growth of the factories, *the height of the pans was increased and steam coils added. Still later the steam jacket was dispensed with, and the steam coils enlarged and extended.* Unfortunately the effect of the coils was miscalculated, and *high-pressure steam* had to be used as a source of heat.

Summary.—Such are the vacuum pans of the present day. We shall see later on that opinions have changed, and that more rational vacuum pans have been constructed for a few years back.

According to Hausbrand (*Evaporating*, etc., p. 61), in a vacuum pan with 650 millimetres vacuum there are evaporated in one hour per square millimetre of heating surface—

By exhaust steam at 110° C.—

From water	100 to 110 litres.
„ thin liquors	60 „ 70 „
„ thick liquors	30 „ 45 „

By high-pressure steam at 130° C.—

From water	130 to 175 litres.
„ thin liquors	80 „ 100 „
„ thick liquors	40 „ 55 „

All that has just been enunciated, with the object of explaining the nature of *single-effect evaporation*, is a necessary preliminary to the study of *multiple-effect plant*; but, as already mentioned, the study of Howard's vacuum pan will be resumed under Sugar Refining, p. 357.

Multiple-effect evaporation plant in vacuo.—Pecquer's system.—Principles of his plant.—Attempts had indeed been made, before Rillieux's successful attempt in 1830, to construct multiple-effect concentration systems, *under pressure*, for the economical evaporation of chemical products. These vessels were constructed on the principles of Pecquer, as follows. A boiler was heated by a furnace to a *high pressure*. The pressure thus produced was used to heat a second boiler at a *lower pressure*, containing liquor coming from the first boiler. This second boiler heated a third under *still further reduced pressure*, and so on until the last, which boiled in the open air. The liquid circulated from the first into the second, and then into the third, into the fourth, and so on.

The theory of multiple effect without vacuum.—The advantage of such an arrangement is easily seen. If 1 lb. of coal yields 5 lbs. of steam in the first pan, these 5 lbs. of steam, condensing on the heating

surface of the next pan, will there evaporate other 5 lbs. of steam. These second 5 lbs. will again evaporate still another 5 lbs., and so on, in such a way that with four consecutive pans with 1 lb. of coal 20 lbs. of water would be evaporated.

The inherent defects of Pecquer's system.—This method of working is known as *evaporation by multiple effect*, as applied to sugar. But from what has just been said it will be easily seen that such a plan would not be at all suitable for sugar factories, because the sugar would be rapidly spoiled under the excessive pressure which such a plan would entail; for if the last pan boils at 1 atmosphere (212° F.), the one next to it would boil at 2 (250°·52 F.), the other at 3 to 4 (285°–294° F.), and the first at 5 atmospheres (309° F.). But sugar juice brought to a temperature corresponding to 5 atmospheres (309° F.) is immediately destroyed.

Differentiation of the Rillieux system from that of Pecquer.—Pecquer's system could not therefore be used in sugar factories. It was about this time that Rillieux invented a vacuum-evaporation system, which constituted such a real progress, such a great step in advance in the sugar industry. Instead of, like Pecquer, using as the last unit of the system a vessel boiling at atmospheric pressure, Rillieux used instead a Howard's vacuum pan, which he heated by the steam coming from the juice of another pan, itself heated by low-pressure steam. He thus created a double-effect evaporation system, the second pan working *in vacuo*.

The principle of the Rillieux system of multiple-effect evaporation in vacuo.—The principle of such a system is as follows. The juice, boiling at a very low pressure in the last pan, the jacket of this pan (supposing a Howard's vacuum pan to be used) is in contact with a liquid corresponding to the temperature of the vacuum.

If this comparatively cold surface be used as a condenser for another Howard's vacuum pan, the second pan will boil under a relative vacuum corresponding to the heat of this condenser, but not so great a vacuum as that existing in the pan, which is used as a condenser because cold water is injected into it.

There is therefore a great vacuum in the last pan and a smaller one in the one in front. Under the action of this smaller vacuum the juice will boil at a rather higher temperature, but, nevertheless, lower than if it had been boiled at the atmospheric pressure. It also can therefore be heated with low-pressure steam.

Rillieux therefore used, as a source of steam for this new pan, in front of the last, another similar pan; but this latter pan he heated with virgin steam, which had no need to be of very high pressure, say half an atmosphere above the atmospheric pressure, or 112° C., say 233°·6 F.

Summary.—Thus the triple effect *in vacuo* came into existence, which the inventor, Horsin-Deon tells us, had to safeguard for fifteen

years before he could find a manufacturer intelligent enough to try in actual practice the system which, at the present day, constitutes the basis of all the saving in fuel hitherto effected in sugar factories.

The modifications effected in the vacuum pan so as to convert it into a triple-effect vessel.—In order to apply his triple-effect system in actual practice, Rillieux made a series of alterations in the vacuum pans. Howard's pan had only a very limited heating surface; to construct an evaporating system for beet-sugar factories from similar surfaces would have been tantamount to making a plaything. As the quantities of juice are so very bulky compared with the volume of syrup to be boiled, Howard's vacuum pans were but barely sufficient, even for the syrup itself alone.

As a matter of fact, 80 per cent. of the juice has to be evaporated before it is made into syrup, and 50 per cent. of the syrup before it becomes crystallisable. The ratio is therefore great. (See also Table XI. p. 178.)

Rillieux preferably imparted the form of a locomotive boiler to his triple-effect units.—Rillieux, in his efforts to find a form of pan presenting a large heating surface, found no better shape to apply to his triple-effect units than the form of a locomotive boiler, which perfectly answered his purpose. The juice was in the boiler, and the steam circulated in tubes.

Description of the various units which enter into the triple-effect system.—The triple effect was accordingly built up in this way. Three locomotive boilers were placed side by side. Steam at half-atmosphere was run into the first pan and caused the juice in it to boil. The steam of the juice rose up into the dome of No. 1, and entered by means of a large pipe into the tubes of No. 2. The steam formed in No. 2 heated No. 3 in the same way, and the steam issuing from the dome of No. 3 went to the condenser and the air-pump. The function of this pump was to extract the air as well as the water which flowed into the condenser.

Safety vessels to prevent frothing and priming.—As the juice froths much, there is much sugar carried over. In order to recover this entrained sugar, Rillieux invented safety vessels (T, Fig. 46A)—boxes fixed on the course of the pipe so as to slacken the speed of the steam, and to allow it time to deposit the drops carried over. This vesicular carrying over of juice by the steam will be dealt with later on. It being advantageous to be able to see what is going on in the evaporation vessels, Rillieux made a small aperture (Fig. 46B) in the front of the boilers and inserted a pane of glass.

Utilisation of exhaust steam from engines to heat triple effect.—Again, sugar factories have a certain number of steam engines—amongst others the air-pump engine. As the steam which escapes from these engines possesses all its latent heat, and as it is possible to impart a slight counter pressure to the engines without injuriously

affecting their working, Rillieux, instead of letting all this steam escape on to the roof, utilised it to heat the No. 1 vessel of his triple effect, thus economising all the waste steam from the engines hitherto completely lost. A little direct steam from the engine boilers was added thereto if the engines themselves did not supply enough for the purpose.

Costly nature of the copper vessels previously in use.—Before the practical application of Rillieux's system all the evaporating pans were made of copper, which made them very costly. It was Rillieux, again, who was the first to substitute cast iron and wrought iron for

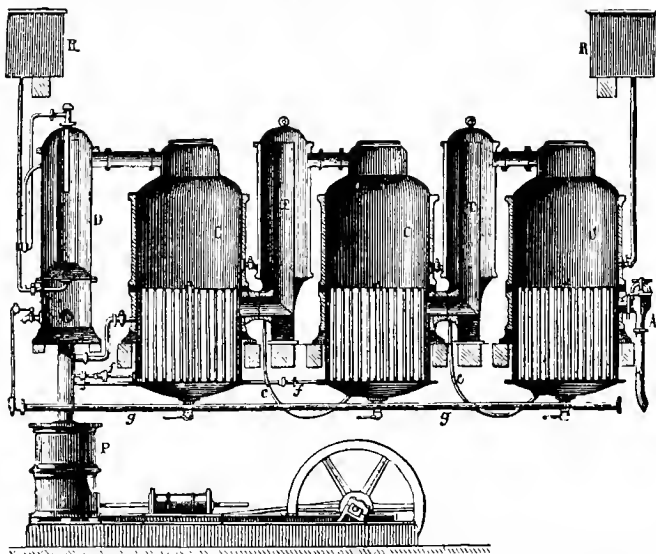


FIG. 46A.—Triple-effect evaporation plant. Showing internal arrangements of No. 1, No. 2, and No. 3 vessels C C C, with their sugar recoverers T T, condenser D, and pump P (C.A.I.L.).

copper in the construction of evaporation plant, having first of all previously ascertained that no damage was occasioned to the sugar, thus effecting a great economy on their prime cost.

Change from horizontal to vertical position the only change since initiation.—Little or no change has been effected in the original triple-effect plant, except to change them from the *horizontal* to the *vertical*. In Austria, however, there would appear to be a tendency to revert to *horizontal* vessels, whilst certain German and American makers of this class of machinery have never deviated from Rillieux's original type.

The perfect nature of Rillieux's system demonstrated by the few improvements yet brought to bear on it.—We are therefore dealing with an evaporation system which for over fifty years has not been improved upon: the only thing which differentiates the plant of the present day from that originally used is the progress made by the boiler makers in the construction of the metallic body of the plant. In Louisiana, U.S.A., where the first sets of this evaporation plant

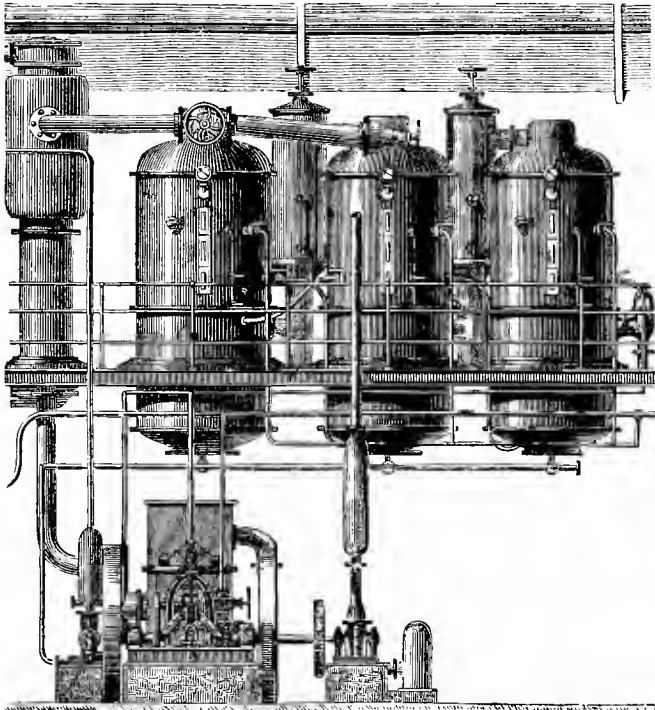


FIG. 46B.—Triple-effect evaporation plant. Showing No. 1, No. 2, and No. 3 vessels with their sugar recoverers, condenser, and air-pump (CAL).

were built, there are still in existence at the present day original Rillieux triple-effect plant at work, quite as well constructed as those now made, and giving the same turn-out and useful effect.

Rillieux triple effect with supplementary vacuum pan misleads the European constructors of triple-effect plant.—Rillieux, moreover, had made an addition to his triple-effect apparatus in the shape of a vacuum "strike" pan, on the same plan as the evaporating vessels

themselves, but the number of tubes in it was diminished by one half. This pan was heated by the steam coming from No. 1, in such a way that No. 1 heated simultaneously both No. 2 and the vacuum strike pan. It was, moreover, placed alongside No. 3, which was thus parallel with it, and its condenser was common to it and the triple effect.

This arrangement, which was adopted with a view of effecting additional economy in steam, as will be explained in the sequel, led to unforeseen and curious results by leading astray those who adopted Rillieux's system in Europe without the concurrence of the inventor.

As a matter of fact, the plans which Tischbein, the German constructing engineer, obtained consisted only of two evaporating vessels and the vacuum strike pan, and Tischbein was so ill informed as to the nature of the plans in his possession that he took the third vessel for one of the series of evaporating vessels, and the Cail Engineering Co., who bought the plans from Tischbein, were of the same opinion. Then Cail, patenting this plant, wrote in his specification that if No. 1 was 100 metres, No. 2 should be 150, and No. 3 of the triple effect should be at least 200 metres.

Numerous discussions occurred over this point, some writers going so far as to declare that it was perfectly evident that Rillieux knew nothing about the system which he had invented.

Matters would have remained so if Rillieux had not returned, some thirty years afterwards, to France, to teach the sugar manufacturers the true principles of the working of the multiple-effect system, and to correct the mistakes committed by the Cail engineers, which led to a loss of 30 per cent. in the turn-out or useful production.

Principles of multiple-effect evaporation.—Summary of facts.—In a multiple-effect system, No. 1 vessel is heated by exhaust steam from the engines, with the aid, if need be, of virgin steam, whilst the steam from No. 1 juice heats No. 2, whilst No. 2 steam in its turn heats No. 3, and the steam from the latter passes to the condenser and air-pump. When a vacuum is made in No. 3 there is a smaller vacuum made in No. 2, and a still less in No. 1, so that, without having to use an air-pump in each vessel, by placing one at the end of the series the vacuum is transmitted backwards in a gradually diminishing form until it reaches No. 1.

Total fall.—Definition.—Taking the steam heat to be at 112° C. (233°·6 F.), and if the vacuum corresponds to a boiling temperature of 60° C. (140° F.), the *total fall* of the temperature of the vessel will be the difference $112 - 60 = 52^{\circ}$ C. (125°·6 F.). It is this difference between the initial and final temperature of the vessel which causes it to boil, and the greater the fall the more energetically does the liquid boil. It is therefore very desirable to utilise collect-

ively all the means possible to increase this fall. It may be as well, however, to mention that Hausbrand is of opinion that the amount of evaporation effected in No. 1, and also the actual consumption of heating steam, are not to any considerable extent proportional to the fall in temperature.

Hausbrand also comes to the following conclusions as to the division of the heating surfaces in the most efficient manner :—

1. The smallest amount of heating steam required to produce a certain amount of evaporation is used in all multiple evaporators when the fall in temperature is the same in each vessel.

2. However the fall in temperature in the separate vessels be arranged, the weight of heating steam to be supplied to No. 1 always varies within very narrow limits. Thus the manner in which the available fall in temperature is distributed amongst the separate vessels has no great influence on the economy of steam. No considerable saving in steam can be obtained by any definite division in this fall of temperature.

3. The quantity of water to be evaporated in No. 1 is on an average of the total evaporation of the multiple evaporator :—

$$\text{In the double effect } \frac{1}{2 \cdot 147} = 0 \cdot 466 \quad D_1 = (W - U)0 \cdot 466.$$

$$\text{In the triple effect } \frac{1}{3 \cdot 333} = 0 \cdot 300 \quad D_1 = (W - U)0 \cdot 300.$$

$$\text{In the quadruple effect } \frac{1}{4 \cdot 626} = 0 \cdot 216 \quad D_1 = (W - U)0 \cdot 216.$$

The extreme cases are—

$$\text{For the double effect } \quad \cdot \quad D_1 = (W - U)0 \cdot 434 \text{ to } 0 \cdot 484.$$

$$\text{For the triple effect } \quad \cdot \quad D_1 = (W - U)0 \cdot 2777 \text{ to } 0 \cdot 3152.$$

$$\text{For the quadruple effect } \quad \cdot \quad D_1 = (W - U)0 \cdot 1926 \text{ to } 0 \cdot 2335.$$

4. The evaporation effected by heating is in all cases the least in No. 1, but the increase in the following vessels is not very great—at most 4 per cent. In the mean it may be assumed that this evaporation in the separate vessels is in the—

As—	Double effect.		Triple effect.			Quadruple effect.			
	No. 1.	No. 2	No. 1.	No. 2.	No. 3.	No. 1.	No. 2.	No. 3.	No. 4.
	d_1	d_2	d_1	d_2	d_3	d_1	d_2	d_3	d_4
	1	1·045	1	1·01	1·04	1	1·005	1·012	1·02

5. The total quantity evaporated in the last vessel is—

$$\text{In the double effect } \quad \cdot \quad \cdot \quad \cdot \quad 0 \cdot 534$$

$$\text{In the triple effect } \quad \cdot \quad \cdot \quad \cdot \quad 0 \cdot 3703$$

$$\text{In the quadruple effect } \quad \cdot \quad \cdot \quad \cdot \quad 0 \cdot 284$$

of the total evaporation of the apparatus (W - U).

XLV.—MULTIPLE EFFECT (JELINEK).

	Simple Effect.		Double Effect.			Triple Effect.			Quadruple Effect.				Quintuple Effect.				
	I.	II.	I.	II.	III.	I.	II.	III.	IV.	I.	II.	III.	IV.	V.			
1. $t_2 - t_1$ = Difference of temperatures t_1 .*	52	26	17.3	17.3	17.3	13	13	13	13	13	10.4	10.4	10.4	10.4			
2. t_2 = Temperature of the heating steam	112	86	112	94.6	77.4	112	99	86	73	60	112	101.6	80.8	70.4			
3. t_1 = Temperature of the boiling juice	60	86	60	94.6	77.4	60	90	86	73	60	101.6	91.2	80.8	60.0			
4. Indication of manometer (millimetres)	008	304	608	112	442	608	27	300	434	608	†	207	398	608			
5. Absolute pressure in millimetres	152	450	152	648	318	152	733	460	266	152	804	553	367	152			
6. Kilogrammes of steam condensed per hour and per square metre of surface	129.7	64.98	61.50	43.35	40.81	41.16	32.52	30.54	30.73	30.92	26.01	24.40	24.51	24.73			
7. Kilogrammes of water evaporated per hour and per square metre of surface	124.8	61.50	62.28	40.81	41.16	41.50	30.54	30.73	30.02	31.11	24.40	24.51	24.60	24.74			
8. Coefficient of transmission of heat—Jelinek's plant	22	22	22	22	22	22	22	22	22	22	22	22	22	22			
9. Heated surface established theoretically, that of the first vessel = 100m ² †	100	100	97.84	100	96.2	99	100	95.5	98	100	100	95.1	96.8	98.7			
10. 1 Kilogramme of steam entering the first vessel evaporates kilogrammes of water	0.9	1.96	2.85	3.79	4.72	5.13	5.3	5.8	6.38	6.8	7.18	7.5	7.8	8.1			
11. Kilogrammes of steam necessary to evaporate 100 of water from the juice	111.11	51.3	35.08	26.38	21.18	15.2	11.2	8.6	6.7	5.2	4.0	3.1	2.4	1.9			

* The temperature of the entrance juice being = 75° C.

† 44 millimetres above the atmospheric pressure.

* Number of vessels.

† Under pressure.

Coefficients of transmission of heat (H. Jelinek).—The number of calories of heat transmitted in a minute per square metre of heated surface and per degree Centigrade of difference of temperatures—

Heating the 1st carbonatation	.	.	C = 3·47
„ 2nd and 3rd	.	.	C = 7·62
„ filtration	.	.	C = 7·85
To the entrance of the juice into the first vessel of the evaporating apparatus	.	.	C = 9·65
„ evaporation (Wellner-Jelinek system)	.	.	C = 2·2
„ heating the syrup	.	.	C = 4·45

Theoretical calculation of evaporating apparatus; it being given that—

t_0 = Temperature of juice entering the apparatus.

t_1 = Temperature of juice boiling in the apparatus.

t_2 = Temperature of steam entering into the heating chamber.

C = Coefficient of transmission of heat per square metre of surface in one minute, for $t_2 - t_1 = 1^\circ$ C. This coefficient is 22 calories in the case of Wellner-Jelinek's plant, and 15 in Roberts' system.

S = Heated surface of the evaporating plant.

Q = Quantity of heat transmitted in one minute by the heated surface of the plant, say = $SC(t_2 - t_1)$.

λ = Calories necessary for transforming 1 kilogramme of water at 0° C. into steam of t_1 , say = $(606\cdot5 + 0\cdot305t_1)$.

r_2 = Latent heat disengaged by the condensation of steam, say = $(607 - 0\cdot708t_2)$.

Pv = Weight of the condensed steam.

Pe = Weight of the water evaporated by the whole surface of the plant.

Without taking into account the small losses occasioned on cooling, it will be seen when $Q = Pvr_2$, we get—

$$Pv = \frac{Q}{r_2};$$

$$\text{and if } Q = Pe(\lambda - t^\circ), \text{ we get } Pe = \frac{Q}{\lambda - t_0}.$$

$$\text{Hence } Pe = \frac{SC(t_2 - t_1)}{606\cdot5 + 0\cdot305(t_1 - t_0)}.$$

To simplify calculation, we shall substitute for the evaporation of the saccharine juice that of water, and increase the heated surface of each of the vessels of the plant by 4 per cent., so as to make allowance for the different boiling-points of the two liquids.

Example I.—*Calculation of a simple-effect plant.*

Let us admit that—

$$t_0 = 75^\circ \text{ C.}$$

$$t_1 = 60^\circ \text{ C.}$$

$$t_2 = 112^\circ \text{ C.}$$

$$t_2 - t_1 = 52^\circ \text{ C.}$$

$$S = 100 \text{ square metres.}$$

$$C = 22 \text{ calories.}$$

$$Pv = \frac{114,400}{607 - 0.708 \times 112} = 216.3 \text{ kilogrammes per minute.}$$

$$Pe = \frac{114,400}{(606.5 + 0.305 \times 60) - 75} = 208 \text{ kilogrammes of water.}$$

That is to say, that the simple-effect apparatus, having 100 square metres of heated surface, evaporates per minute 208 kilogrammes of water with an expense of 216.3 kilogrammes of steam.

A square metre of surface therefore evaporates 124 kilogrammes of water per hour, and 1 kilogramme of steam evaporates $\frac{208}{216.3} = 0.96$ kilogrammes of water.

Example II.—*Calculation of a double-effect plant.*

No. 1 Vessel—

$$t_2 - t_1 = \frac{52}{2} = 26^\circ \text{ C.}$$

$$t_2 = 112^\circ \text{ C.}$$

$$t_1 = 86^\circ \text{ C.}$$

$$t_0 = 75^\circ \text{ C.}$$

$$S = 100 \text{ square metres.}$$

$$C = 22 \text{ calories.}$$

$$r_2 = (607 - 0.708 \times 112) = 527.7.$$

$$Q = 22 \times 100 \times 26 = 57,200 \text{ calories.}$$

$$(\lambda - t_0) = (606.5 + 0.305 \times 86) - 75 = 557.73 \text{ calories.}$$

$$Pv_1 = \frac{57,200}{527.7} = 108.3 \text{ kilogrammes of condensed steam.}$$

$$Pe_1 = \frac{57,200}{557.73} = 102.5 \text{ kilogrammes of water evaporated.}$$

No. 2 Vessel—

$$t_2 - t_1 = \frac{52}{2} = 26^\circ \text{ C.}$$

$$t_2 = 86^\circ \text{ C. for the second vessel.}$$

$$t_1 = 60^\circ \text{ C.}$$

$$t_0 = 86^\circ \text{ C.}$$

$$r_2 = (607 - 0.708 \times 86) = 546.$$

$$(\lambda - t_0) = (606.5 + 0.305 \times 60) - 86^\circ \text{ C.} = 538.8.$$

$$Q = P v_2 r_2, \text{ or (since } P v_2 = P e_1) = P e_1 r_2 = 102.5 \times 546 = 55,965.$$

$$S = \frac{55,965}{22 \times 26} = 97.84 \text{ square metres.}$$

$$P v_2 = P e_1 = 102.5 \text{ kilogrammes of condensed steam.}$$

$$P e_2 = \frac{55,965}{538.8} = 103.8 \text{ kilogrammes of condensed steam.}$$

A double-effect apparatus, having $100 + 97.84$ square metres of heated surface, can evaporate per minute $102.5 + 103.8$ kilogrammes of water. A square metre of double-effect surface will evaporate per hour only 62.5 kilogrammes of water, by expending 108.5 kilogrammes of steam on the first vessel. A kilogramme of steam will evaporate $\frac{206}{108.3} = 1.9$ kilogramme of water. If the heated surface of the second vessel be less than that of the first, it is because the juice which runs into it is of a higher temperature.

Theoretically, the three vessels of a triple-effect apparatus ought each to possess the same heating surface. However, owing to the formation of encrustations on the tubes of the second, and to a still greater extent on those of the third, the surface of the latter is increased by 10 per cent. per vessel. The triple-effect is therefore so constructed that No. 1 has 150; No. 2, 165; No. 3, 185 square metres of heating surface—say 500 in all. (See table, p. 198.)

PRACTICAL APPLICATION OF FOREGOING DATA

Take the case of a factory working 10,000 kilogrammes of beet-root per hour, and extracting by diffusion 14 per cent. of juice of 9° Balling (dissolved matter), and making syrup of 50° Balling. The evaporating apparatus being triple effect, the quantity of water to evaporate per hour will be—

From the diffusion juice	$\frac{10,000 \times 140}{100} \left(1 - \frac{5}{90}\right)$. = 11,480 kg. (1)
,, milk of lime (= 3 per cent. CaO)	14.9 per cent.	= 1,490 kg. (2)
,, washing filter press sludge, etc.	11	,, = 1,100 kg. (3)
Say in all	.	<u>14,070 kg.</u>

Or in round numbers 14,100, which gives each vessel 4700 kilogrammes of water to evaporate.

In the preceding table (XLV. p. 198) it will be found that in a triple-effect apparatus each square metre of surface can evaporate about 40 kilogrammes of water per hour. Let us admit that, in practice, only 75 per cent. of the theoretical effect is obtained, and we get

$\frac{4700}{40 \times 0.75}$ = about 160 square metres. In the case of apparatus on Roberts' system, the theoretical effect can only be taken as 50 per cent., the coefficient of transmission being only 15 instead of 22, the figure established for the Wellner-Jelinek apparatus. We thus get

$\frac{4700}{40 \times 0.50}$ = about 235 square metres.

- (1) Taking S_1 = dry substance (per cent. gr.) of the juice,
 and K_1 = its weight in kilogrammes;
 Taking S_2 = dry substance (per cent. gr.) of the syrup,
 and K_2 = its weight in kilogrammes;
 Taking E = quantity of water to evaporate,
 we get—

$$K_2 = K_1 - E, \text{ then } K_2 = \frac{K_1 S_1}{S_2}, \text{ whence } E = K_1 \left(1 - \frac{S_1}{S_2}\right).$$

(2) A litre of milk of lime of 19° B., sp. gr. 1.095, contains 193 grammes CaO, and weighs 1152 grammes; it therefore contains 959 grammes of water to evaporate. By using 3 per cent. CaO, say 1554 litres of milk of lime for 10,000 kilogrammes of beets, there will be 1490 litres of water to evaporate.

(3) With 3 per cent. CaO, 11 per cent. of sludge is obtained, which is washed with 100 per cent. of water, which gives 1100 litres of water to evaporate.

JELINEK'S AUSTRO-HUNGARIAN DATA COMPARED WITH SIDERSKY'S FIGURES FOR FRENCH METHODS OF WORKING

Jelinek's calculations apply chiefly to the work of Austro-Hungarian sugar factories, where generally weak juices are evaporated up to 50° Brix = 27°·5 Baumé, sp. gr. 1.253.

In France, as has been pointed out by Sidersky, the work is very variable. There are factories which extract 135 litres of juice of 4°·5 density per 100 kilogrammes of beets, and others which only extract 125 litres of juice with a density of 5°. In average work with beetroots of 7° density, 130 per cent. of juice with a density of 4°·7 may be relied on, say 136 kilogrammes of juice with 11.8 of dissolved substances.

The quantity of water to evaporate to get a syrup of 25° Baumé (= 45°·4 Brix), sp. gr. 1.206, will therefore be $100 \left(1 - \frac{11.8}{45.4}\right) \times 1.36 = 100.64$ kilogrammes.

There is added 10 per cent. of milk of lime of 20° Baumé, sp. gr. 1.16, say 13 per cent. of the beetroots. This milk of lime contains 206 grammes CaO per litre and 956 grammes of water, say 2.64 kilogrammes per cent. CaO and 12.50 per cent. water on the

beetroot. These 2·64 kilogrammes of CaO produce 10 kilogrammes of sludge, which is washed with double its weight, say 20 kilogrammes of water, from which must be deducted 12·50 kilogrammes which return to the source and to the stirring up of the lime. The quantity of water to evaporate for 100 kilogrammes of beetroots will therefore be—

	Kilogrammes.
From the diffusion	100·64
From the milk of lime	12·50
From the washings of the sludge in the filter presses	7·50
	120·64

This gives 12,064 kilogrammes of water to evaporate per 10,000 kilogrammes of beetroot, instead of 14,070 kilogrammes indicated by M. Jelinek. That gives 4021 kilogrammes of water for each vessel per hour, hence the heated surface = $\frac{4021}{40 \times 0.75} = 134$ square metres for the horizontal apparatus of Wellner-Jelinek, or 200 square metres for apparatus on Roberts' vertical system.

As a general rule, it is acknowledged that the heated surface required for evaporation should be 0·13 to 0·15 square metres per hectolitre of juice extracted by diffusion. The Cail Engineering Co. allows the following dimensions for its apparatus:—

	Square Metres of Heated Surface.
For 1800 hectolitres of juice	292
For 2200 " 	360
For 4000 " 	469

EFFECTIVE HEAT OF COMBUSTION OF FUEL—AVERAGE RESULTS FROM BOILERS IN SUGAR MANUFACTORIES

In regard to the efficiency of the boilers used in beet factories, Schmidt has found, when the working pressure is between $4\frac{1}{4}$ and $5\frac{1}{2}$ kilogrammes per square centimetre, say 65-70 lbs. to the square inch, and with a feed-water at a temperature between 81° and 95° C. (177°·8 and 203° F.), there is generally burned in a sugar-works per hour per square metre (3·28 feet) of grate a quantity of coal varying between 67 and 93 kilogrammes, sometimes going beyond 100 kilogrammes. With coals giving $5\frac{1}{2}$ to 8 per cent. of ash there is a waste in the furnaces of $9\frac{1}{2}$ to $13\frac{1}{2}$ per cent., and there is thus left in the cinders between 2 and 7 per cent. of combustible matter. There is lost in the smoke between 17 and 30 per cent. of the heat of the coal. The undetermined loss—that is, exterior radiation, soot, and imperfect combustion—represents, including the carbon in the ashes, between 6 and 10 per cent. of the heat of the coal. The remainder

represents the heat utilised, which, in even numbers, varies between 64 and 70 per cent.

This corresponds to a production of steam varying between 9 and $9\frac{1}{2}$ lbs. of steam per lb. of coal burnt, under the above conditions of temperature of water and pressure of steam. This amount of steam represents variations between 11 and 16 kilogrammes per hour per square metre of heated surface, between 200 and 300 kilogrammes per hour and square metre of the plane of water, between 400 and 800 kilogrammes per hour and cubic metre of the steam chamber. With coals costing 20 to 21 francs per metric ton delivered at the factory, the 1000 kilogrammes of steam are obtained at prices varying between 2 and $2\frac{1}{4}$ francs. Exceptionally, in the case of coals with heavy freight charges delivered at 30 francs (£1, 4s.) the ton at the factory, the price of the ton of steam exceeds 3 francs (say 2s. 5d.). In 1895 about 1000 kilogrammes of steam were consumed per ton of beetroot; at the present time it has been found possible to diminish this consumption in certain factories. Working expenses have decreased in proportion.

According to Schmidt, all the steam boilers used in beet-sugar factories are good if well proportioned. The feed-waters in beet-sugar factories require the use of tubular boilers. But semi-tubular boilers may give as economical results as tubular ones of the locomotive class, by adopting heated surfaces provided with restraining bars, which practically limit the production of steam.

The rational regulation of the initial and final temperatures.—*High-pressure steam darkens the juice.*—In order to increase the falls, it might be attempted to increase the initial temperature and diminish the final temperature. However, for example, if the pressure of the initial heating steam be increased so as to raise the temperature from 112° to 118° C. ($233^{\circ}\cdot6$ to $244^{\circ}\cdot4$ F.), the fall is no doubt increased, but there is reason to believe that steam at this temperature would injuriously affect the juice. Accordingly the steam is recommended by Horsin-Deon to be kept at a temperature of 112° C. ($233^{\circ}\cdot6$ F.), corresponding to half atmosphere above the ordinary atmospheric pressure, going at times as high as 118° C. ($244^{\circ}\cdot4$ F.), but never exceeding the latter, beyond which the juice, without being spoilt so far as sugar is concerned, might be blackened by the decomposition of organic matter, unless under those special conditions of rapid circulation which are sometimes met with in the vessels called circulators. As to the pressure in the last vessel, it can be reduced to a temperature of 60° C. (140° F.), or 55° C. (131° F.), or even to 50° C. (122° F.), without any risk. In fact, the lower the temperature the better is the condition of the syrup maintained. This desirable result is obtained by increasing the power of the condensers and air-pumps. The maker should pay most attention to this point, but unfortunately he does not give it the attention which it deserves.

Horsin-Deon's formulæ and deductions.—If we take—

m = the weight of the condensed steam per unit of surface and fall of temperature,

t = temperature of heating steam,

t_1 = temperature of boiling liquid,

it follows that on a surface S there will be condensed—

$$Q = mS(t - t_1).$$

Application of above formulæ.—The quantity of steam condensed (m) is given by the formula which we have enunciated (pp. 187, 188). To obtain a given result, t and t_1 depend on the installation of the system, and are known.

There only remains undetermined the value of S , which may be deduced from the above equation, as also can the heating surface of a system whose principal elements as named above are known.

If t be the temperature of the heating steam, and t_1 the temperature corresponding to the vacuum given by the pump, what will be the proportional surface to give to the different vessels of a simple effect, double effect, and triple effect? As far as a simple effect is concerned, the above formula gives the required surface straight away.

Case of equal distribution of the fall between No. 1 and No. 2 vessel.—In the case of a double-effect system, supposing in the first place that the total fall is spread over each of the two vessels, this distribution may be effected equally over each of the two so that the falls will be $\frac{t - t_1}{2}$ for each vessel.

Equal evaporative effect of No. 1 and No. 2.—Moreover, it is known that No. 1 vessel, causing No. 2 to boil, the same amount of steam is formed in No. 1 as in No. 2, because, if more steam were present in No. 1 than in No. 2, this excess could not be utilised, which is impossible; whilst, if a smaller quantity were formed, No. 2 could not evaporate more than No. 1 caused it to do. Consequently the evaporation in each of the vessels will be $\frac{m}{2}$.

If S_1 and S_2 be the surfaces of the two vessels, the quantity Q of condensed steam will be—

$$Q = S_1 \times \frac{m}{2} \times \frac{t - t_1}{2} + S_2 \times \frac{m}{2} \times t - t_1 = (S_1 + S_2) \left[\frac{m \times t - t_1}{2} \right].$$

As in the single-effect system the same amount of work is done with the surface S , we get—

$$Sm(t - t_1) = (S_1 + S_2) \frac{m(t - t_1)}{2}.$$

$$\text{Hence } 2S = S_1 + S_2.$$

Ratio of the area of the heating surface of the single-effect system to that of the double-effect system.—The heating surface of the double-

effect system ought therefore to be double the size of the surface of the single effect ; and as S_1 and S_2 have the same work to do, with the same fall, they should therefore be equal, and the double effect should consist of two equal vessels, each having the same surface as that which a single-effect system would have, working in the same conditions.

The evaporative power is not increased by adding more units.—In the same way it could be demonstrated that the surface of the triple effect should be $S + S + S$. In a quadruple-effect system there would be four vessels equal to S , and so on. Therefore when a vessel equal to the others of the series is added to a multiple-effect system, which by this addition is converted into a system of higher multiple effect, the evaporating power of the system is not increased.

The benefit lies in the less amount of steam required to heat the system, but advantages diminish progressively after the first addition.

—But the quantity of steam required to heat the system is diminished, because with a single-effect system m kilogrammes of steam required to be applied, in the double effect $\frac{m}{2}$, in the triple effect

$\frac{m}{3}$, then $\frac{m}{4}$, and so on. But as the difference between m and $\frac{m}{2}$

is $\frac{m}{2}$, the difference between $\frac{m}{3}$ and $\frac{m}{4}$ is $\frac{m}{12}$, it will be seen that the passage from the single effect to the double effect produces six times as great an economy as the passage from the triple effect to the quadruple effect, and that the passage from the quadruple effect to the quintuple effect only effects an economy of $\frac{m}{20}$.

The higher the multiple the less the economy effected by another addition.—The greater the departure from the single effect, therefore, the less is the economy obtained by adding another unit to those pre-existing ; and before changing from one system to another it should be ascertained whether the resultant economy is worth the trouble of increased plant,—whether, in fact, the economy realised would pay capital and interest for the new plant purchased. *It has been found in actual practice that the quadruple effect is the extreme limit to employ, the quintuple effect not paying interest and capital on the additional vessel.*

The comparative weight of steam to be condensed in single- and triple-effect plants, and the effect thereof on the capacity of the air-pump.—In a simple-effect plant the amount of steam to be condensed is m , in a double effect $\frac{m}{2}$, in a triple effect $\frac{m}{3}$, etc. Therefore the greater the multiple effect the less will be the amount of condensation water necessary, and the smaller will be the air-pump required. That is also a point worthy of notice.

The variation of the "falls" from vessel to vessel in multiple-effect systems.—We have taken it for granted at the outset, that, in order to ascertain the proportions to be given to the vessels of an evaporation system, the *falls* were equal between each vessel. In reality, however, this is not so. The falls, far from being equal, keep on increasing from the first to the last vessel. Thus in a triple effect, with vessels of equal size, the falls are 9°, 12°, 18° C. between each of the vessels.

This variation formerly attributed to faults of construction.—This fact had escaped the notice of those who attributed the differences which they had observed to some fault in construction of the plant.

The variation in pressure causes the same weight of steam to vary from vessel to vessel.—The vacuum being different in each vessel, the same weight of steam occupies a different volume, and this volume increases along with the vacuum from the first to the last vessel.

The density of steam as affected by a greater or less vacuum, and influence thereof on rapidity of condensation.—If we consider the unit of weight of steam per unit of condensing surface, the former will form a deeper layer the greater the vacuum. Consequently a molecule of steam placed on the surface of the layer will have a greater space to travel before reaching the condensing surface, in proportion to the greater thickness of that layer, where the vacuum is more perfect. Therefore it will take longer time to traverse this distance, according to the formula $e = \frac{1}{2} gt^2$.

$$\text{Hence } t = \sqrt{2 \frac{e}{g}}$$

If the molecule takes longer to traverse the distance which separates it from the condensing surface, the condensation will be retarded proportionately in the same time.

Therefore, with the same fall of heat the last vessel will, in the same space of time, condense less steam than the first. Moreover, as, in order that the plant may work properly, it is indispensable that each of the vessels condense the same amount of steam, it follows as a matter of course that the last vessel requires a greater fall of heat than the first.

Let—The heating surface of a vessel = S.

The quantity of steam condensed on that surface = e .

The fall of temperature which causes the liquid to boil = C.

The specific volume of the steam = V.

The difference of pressure which induces the movement of the steam = h .

The coefficient of evaporation special to the apparatus = K.

We get the formula—

$$e = \frac{ScK \sqrt{2gh}}{V}$$

which gives the evaporation of a vessel of which all the data are known, hence—

$$K = \frac{e}{Sc} \times \frac{V}{\sqrt{2gh}}$$

which gives K in a known evaporation plant. K is constant in all triple-effect plant, with steam at the same temperature, and varies with the temperature of the steam used.

The use and applications of above formula.—All the various phenomena met with in multiple-effect systems may be explained by means of this formula, and the proper falls of heat which ought to occur between each vessel calculated from it.

Effective condensation only one item in efficient working of triple-effect systems.—But the condensation of steam is only one of the items which affect the working of a multiple-evaporation system.

Incidental phenomena.—When the steam is condensed it transmits its latent heat to the condensing surface, and this surface transmits it, in its turn, to the boiling liquid, and this liquid finally passes into the form of steam.

Necessity for combining the various formulæ into a single comprehensive one.—All these successive phenomena would have to be taken into account and formulated, and all these formulæ combined in a single comprehensive formula. This formula, however, is very complex, and, moreover, cannot be applied to the current working of evaporation plant.

The results arrived at.—It has, however, been used to establish the relative proportions which the different vessels of a triple-effect system should bear to each other, with the simple result, after a lengthened analysis, that all the vessels should be equal, as shown in the calculations previously enunciated, provided always that the transmission of heat through the condensing sides be the same in each vessel.

The effect of variations in heat conductivity due to encrustations.—But, again, for special reasons outside the domain of physics, the conductivity of the metal tubes is continually varying. This is caused by the encrustation which is formed on the tubes. The juice contains mineral and calcic salts, more soluble in the juice than in the syrup, which are deposited on the tubes, in very hard layers, in proportion as the liquid becomes concentrated to such an extent that the tubes of the last vessel are covered with a thicker crust than the preceding, which itself contains more than the one in front, and so on.

The encrustations become so dense as to stop work.—As these encrustations are very bad conductors of heat, there eventually comes a time when the layer has reached a certain thickness, variable with the nature of the deposit, when the evaporation plant will not work. It must therefore be cleaned.

Suggested possible advantage in progressively increasing theoretical surface.—But, so long as this extreme point is not reached, it is evident that the conductivity for heat is different in each vessel, getting from bad to worse from the first to the last vessel. There is therefore, they say, some advantage in increasing the theoretical surface of the vessels most liable to encrustation, and to make a so-called differential progression in the surfaces of each of the vessels.

The apparent fallacy of such a course.—But this progression is nothing but a mistake, and is even useless; evaporation goes on in spite of these changes in conductivity, only the differential progression is then effected naturally, not on the surfaces which remain constant, but on the essentially variable falls of temperature in each vessel, which follow, moreover, the physical laws corresponding to the changes in the conductivity of the condensing surfaces. There is no advantage, therefore, in constructing differential vessels.

Uniform size of the units the most rational course to adopt.—In all rational multiple-evaporation systems, therefore, the vessels are equal, and the falls of temperature between each vessel are differential, and regulated by the law of progression of specific volumes of vapours, and the diminution in the conductivity of the metal through encrustation.

Influence of density on boiling-point.—To these it is still necessary to add that the greater the density of the sugar liquors the higher is the temperature at which they boil. (See Table XLI. p. 182.)

Effect on the "falls."—Consequently the falls increase proportionately from the first to the last vessel, with the amount of this increase in the boiling temperature of the liquid being evaporated.

Hausbrand treats the theory of multiple-effect evaporation in a somewhat different form from Horsin-Deon, but the reader must be referred to the manual of the former on *Evaporating*, etc., pp. 62–116 for details. It must suffice here to state that, with the aid of abstruse calculations, diagrams, formulæ, and tables, he solves the following question, with the result given below.

Example.—100 litres of liquor are to be evaporated to 10 litres in a quadruple-effect evaporator, in the elements of which the temperatures 100°, 95°, 85°, and 50° C. are maintained. How much water is evaporated in each vessel? The answer given by Hausbrand is—

	No. 1.	No. 2.	No. 3.	No. 4.
	Litres.	Litres.	Litres.	Litres.
Regular evaporation	20·655	20·731	20·850	21·26
Self-evaporation .	0	0·7325	1·787	3·952

$$\text{Total . . . } 20\cdot655 + 21\cdot4635 + 22\cdot637 + 25\cdot212 = \left\{ \begin{array}{l} 89\cdot9875 \text{ litres} \\ \text{of water.} \end{array} \right.$$

CLASSEN'S CONCLUSIONS FROM HIS EXPERIMENTS ON VERTICAL
MULTIPLE-EFFECT APPARATUS

1. That the coefficient of transmission, and consequently the evaporative power, with a small height of juice, is from 20 to 30 per cent. greater in the No. 1 and No. 2 vessels, and from 10 to 20 per cent. greater in the No. 3 vessel than with a greater height of juice.

2. When working normal beetroots, with the apparatus clean and in good working order, this coefficient remains constant for several months.

3. This coefficient diminishes with the density of the juice.

4. It is greater the greater the pressure of the steam.

5. It diminishes proportionately with the height of the vacuum.

6. It is lower in the last vessel than in the preceding ones, even in those which have a greater heating surface. It follows from these important considerations on the calculation of the power and dimensions of evaporating apparatus, that the calculations hitherto made, with the exception of those of Horsin-Deon, are based upon the incorrect supposition that the coefficient of transmission is constant in all the vessels. Jelinek acknowledges that this supposition is false, but up to now has come to no conclusion.

In fact, it is only so far exact as to determine the surface of No. 1 vessel, and to give to it the greatest possible fall of heat. On the other hand, the last vessel may be smaller than the others when it is desired to attain the maximum of power. Again, we can at 100° C. still have an evaporation, with a fall of 3° to 4° C., and, without exceeding a counter pressure of 0·5 to 0·6 atmosphere, connect a fifth and even a sixth vessel to the vertical tube apparatus.

Necessity for uniformity in trials made with evaporation apparatus (H. Classen).—The numerous trials made with steam boilers are regulated by well-determined methods. But experiments made with evaporating and boiling apparatus are made without any exact basis, even although the importance of similar tests be very great.

Evaporation experiments may be conducted with two very different objects in view—

1. The determination of the utilisation of the heat of the steam in the apparatus.

2. The determination of the power of the apparatus.

The first point cannot very well be determined; owing to faults in construction of the apparatus, an absolutely exact value cannot be obtained. The measure of the power of an apparatus is the *coefficient of transmission*, i.e. *the quantity of heat transmitted per minute per square metre for one degree C. of difference of temperature*. Data may be obtained, by experiment, from which this coefficient may be calculated. It is necessary that the experiments be made as far as possible under conditions similar to those which will be realised in

actual practice when the apparatus is at work. The points which intervene in establishing the *coefficient of transmission* are—

1. The extent of the heated surface.
2. The quantity of water evaporated or of steam condensed.
3. The duration of the test.
4. The temperature of the heating steam and of the boiling liquid.

1. *Calculation of the heating surface.*—In each heating tube, surface condensation and evaporation must be considered. In vertical tube apparatus, where the liquid circulates in tubes, the surface of condensation is greater than the evaporation surface. The reciprocal takes place in the case of horizontal apparatus and coils. As far as transmission of heat is concerned, the evaporation surface is most important, for the steam condenses on the surface of condensation the moment the temperature is lowered. The interior surface is therefore used for the purpose of calculation in the case of vertical apparatus, and the exterior surface in the case of horizontal tubes and coils. It is, however, better in these tests to always regard the outside surface as the heating surface, and to take only the outside surface, on account of its facilitating calculations, in the case of coils. Uniformity in testing is greatly to be desired in what is known as heating surface. Classen proposes to calculate the surface of a tube by multiplying the inside circumference by the length—that is to say, by the distance between the exterior surfaces and the tubular plates, described on p. 213.

2. *Determination of the water evaporated or condensed.*—In the greater number of instances the liquid entering the apparatus is measured; it follows that the power of the apparatus may be obtained either by measuring the condensed water, or by that of the liquid evaporated. The first should only be used when the steam does not contain vesicular water, or when the vessel, being heated, is served by the vapours of the juice coming from another apparatus; or, in fact, when these two apparatus are very near, and their envelopes as well as their communication pipes are isolated by non-conducting compositions. The second method, based on the amount of condensed steam, may be applied wherever this vapour is condensed, without addition of water. The condensed water is led automatically or pumped into a measuring tank.

3. *Duration of the experiment.*—If the apparatus be working in a regular manner, and the conditions of the experiment vary but little, the test may be limited to a quarter of an hour; but if these conditions be not fulfilled, the experiment must be prolonged.

4. *Determination of temperatures.*—The exact determination of the temperature of the heating steam and of the boiling liquid is of the greatest importance, especially when these temperatures differ, because then each error has a great influence on the amount of the

coefficient of transmission. The temperature of the heating steam is taken as it enters into the steam chamber. The condensed water coming from the steam chamber is always lower in temperature than the steam which has produced it, and this temperature is lower in proportion as the space traversed by the water on the heated surface is longer, and consequently the fall of temperature is greater. The temperature of the water cannot be used to calculate the fall of temperature, as is sometimes done. In exact tests, only the heat contained in the condensed water, corresponding to the temperature of the tension of the steam, should be introduced.

Practically the boiling temperature at the surface is taken. In the boiling of water this temperature is the same as that calculated by the tables which give the tension of steam. But, in causing liquids containing substances in solution to boil, the temperature increases. So as to make a double control of the temperature, use is made of tables for boiling saccharine juices. (See Table XLI. p. 182, etc.)

Definition of the fall of temperature.—It follows from the foregoing that in practical experiments *the fall of temperature should be taken as the difference between the temperature of the steam entering the steam chamber and that of the surface of the boiling syrup.* This fall serves to determine *the coefficient of transmission.* For the determination of temperatures, accurate thermometers agreeing very exactly with one another should be used. They ought, at least, to show $\frac{1}{3}$ of a degree, or, better still, $\frac{1}{10}$. As these thermometers are very costly, and not to be got everywhere, Classen describes a simple method for protecting them. The thermometer is introduced into a tube of thin sheet copper, closed only at one end, and is filled with amylic alcohol, by which the thermometer is surrounded. The heat is thus regularly transmitted. The graduation ought to be very visible.

The manometric determinations cannot be done with the ordinary spring manometers or with vacuum indicators. It is necessary to use mercury manometers, which the operator may construct for himself. It must be observed that all the temperatures and pressures obtained should agree exactly with the tables of the tensions of saturated steam. The barometric and thermometrical readings should be taken simultaneously and repeatedly, so as to get an exact average. Classen then gives the following scheme, which should be followed during testing. This plan is that of an evaporation test, in which the condensed water is measured.

*Scheme or Schedule for the Thermo-dynamical Testing of
Evaporation Plant in Sugar-works*

- | | | |
|---|-----------|-------|
| 1. Heating surface (interior of tubes) in square metres | . | |
| 2. Duration of the test in minutes and seconds | . . | |
| 3. Condensed water in litres | | |

4. Temperature of the condensed water, ° C.
5. Weight of the condensed water
6. Tension of the heating steam in centimetres
7. Temperature of the heating steam calculated, ° C.
8. Temperature of the heating steam measured, ° C.
9. Tension of the juice steam in centimetres
10. Temperature of the juice steam measured, ° C.
11. Temperature of the juice steam calculated, ° C.
12. Degree Brix of the juice in the apparatus
13. Temperature of the entering juice, ° C.
14. Temperature of the boiling juice = temperature of the juice steam + the elevation of the boiling-point, ° C.
15. Fall of temperature, ° C.
16. Water evaporated per square metre per hour in kilo- grammes
17. Water evaporated per square metre per minute for a fall of 1° in kilogrammes
18. One kilogramme of heating steam abandons calories
19. Coefficient of transmission

CONSTRUCTION AND PLAN OF TRIPLE-EFFECT EVAPORATION SYSTEMS

The obsolete double-effect system.—It is unnecessary to speak of the now obsolete *double effect*, which only differed from the treble by the absence of the intervening vessel.

The vertical *triple-effect system*—the harmonious, artistic appearance of which is due to the well-known *Cail Engineering Company* of Paris—consists of three cylindrical vessels (Fig. 46A and Fig. 46B). Each vessel consists of two truncated divisions of about equal height, joined together, the one above the other, by flanges and bolts and nuts. The lower truncated part contains the tubular heating surface (Fig. 46A, p. 194). It consists of a cast- or wrought-iron cylinder 5 to 5½ feet in height, with a diameter varying with the capacity of the vessel. This cylinder is provided at top and bottom with two bronze plates, called tubular plates, pierced with holes of 50 millimetres, say 2 inches, arranged very closely to each other, and in a quincunx fashion on the same vertical as the similar-sized holes on the other, the holes of the one being placed exactly on the same vertical as those of the other, consequently opposite to each other. Into these holes tinned brass tubes, which have the same length as the cylinder, are passed, and firmly fixed therein in such a way that once in their place they do not pass through the plates. These tubes with a few crosspieces keep the two division plates the same distance apart, and thus maintain a tight joint with the cylindrical vessel, which acts as an envelope for them.

Steam enters through a large side tube (see A, Fig. 46A) connected

with the cylindrical vessel, condenses on the outside of the tubes, and escapes as condensed water through a small tap in the bottom of the same vessel on the opposite side to that where it enters.

The calender.—Underneath the lower cylinder known as the tubular vessel, a slightly bomb-shaped bottom is fitted and bolted so as to make a tight joint with it. Above the tubular vessel is the second main trunk already mentioned, called the *calender*.

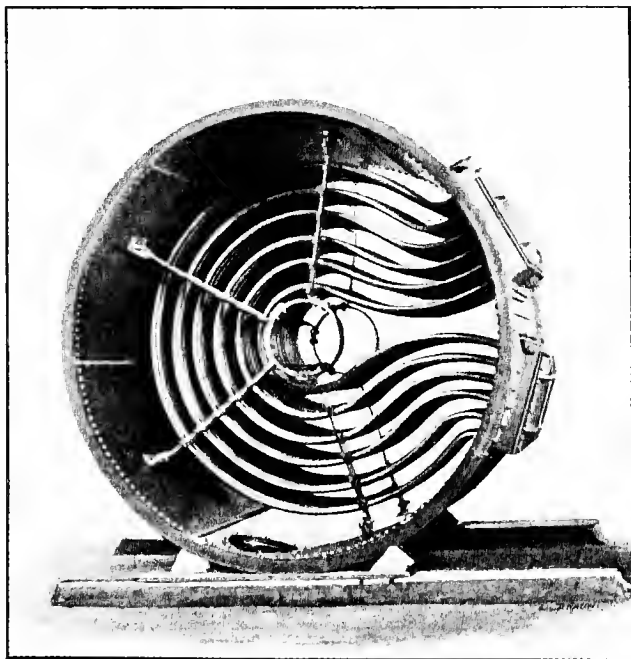


FIG. 47.—Calender of triple-effect vessel, being fitted with steam lyres in the Cail workshops.

Lid.—On the calender is a dome-shaped lid, in the centre of which is a projection through which the steam escapes.

The manner in which each vessel is filled with juice.—The juice is run into the vessel through a tap in the lower part of the calender, almost at the level of the upper tubular plate. The juice flows over the plate and falls through each hole into the tubes, and from there into the lower bottom of the vessel. When that bottom is full the level of the juice rises in the tubes until it overflows and submerges

the upper tubular plate. When that takes place the flow of juice is stopped.

The way in which steam is applied so as to heat the vessels.—Steam is now applied through the lateral tap of the cylindrical vessel. This steam impinges on the *outside* of the pipes containing the cold juice in their interior. It there condenses, and heats the juice to the boiling-point. The juice then evolves steam, which spreads through the calender and escapes through the top of the dome, to be thence conducted, by means of a pipe, into the tubular vessel of an identical cylinder to that just described, and to heat it.

The calender and its accessory arrangements.—The calender (Fig. 46B) has several accessories. First, quite at the bottom, there is a pane of glass fixed on the vessel, so that what is going on inside may be seen. There are generally three of them, one above the other. These panes are long and narrow, and enclosed in a bronze frame. In addition to these, a round glass is placed behind, so as to light up the interior by means of a light, and thus show how the juice is boiling.

The way in which fat is introduced to prevent frothing.—Then comes a *butter* tap, used for introducing fatty matters (the word *butter* is misleading, but it is the term used) so as to beat down the froth which is formed from time to time during boiling, when the juices are not very sound, or if the carbonation has been imperfect (p. 247).

The ammoniacal gas discharge tap.—Finally, besides the juice tap there is a tap for the escape of ammoniacal gases, which is described further on, a water tap to fill the vessel and wash it, a steam valve for the same purpose, and a metallic or mercury pressure gauge.

Sampler.—The last vessel containing the syrup includes a *sampler*, so that a certain quantity of syrup can be extracted as the evaporation proceeds. It is, however, rather a difficult matter to extract liquid from a pan in which there is a vacuum. It is necessary to have a small reservoir (in which the vacuum is the same as that in the pan), communicating with the space containing the syrup by means of a pipe on its top which enters the pan at a higher level than the syrup. In this way the syrup runs into the small vessel. When it is full the vessel is isolated by taps. The air is allowed to enter, and all that has then to be done is to open another tap placed underneath to let the syrup run out. (See Fig. 53, p. 275, and context.)

Safety vessel.—*Vesicular transference of the juice in the path of its vapour.*—When the triple-effect system is boiling, the steam which is evolved carries over with it in its train saccharine vesicles, which are more abundant the more energetic is the boiling and the more concentrated the liquid. This is a fact well known under the title of the Vesicular Transference of Liquids by their Vapours. The word vesicular indicates that the saccharine particle assumes the form of a small hollow sphere, a sort of bubble analogous to a soap bubble, which

is formed in the midst of viscous liquids. In the case of pure water even the vesicle is also produced, but is destroyed as soon as formed. But if the liquid be viscous, like juice, the vesicle persists in the steam for a longer time, and is carried over by it in its train into the pipes. In the case of concentrated syrup the vesicles are so abundant that, if they were not guarded against, a considerable loss would occur through them.

The condensed water is thus contaminated with sugar, which injuriously affects the boilers.—Again, the steam containing the saccharine vesicles is condensed in the following vessel. Therefore the condensed water contains sugar, and this condensed water is used to feed the boilers; sugar is in this way introduced into the latter. Now sugar is the enemy of all boilers. As soon as a certain amount of sugar collects in the boiler the water blackens, forms a froth in the dome, and it is no longer possible to maintain the water-level in the boiler; the indicators “blow off,” the joints disappear, the boiler cracks, and serious accidents are to be feared. The only thing that can be done is to empty all the boilers, the one after the other; such a mishap means a loss of time and money. It is therefore absolutely necessary to prevent the sugar from passing over with the steam into the tubular space of the next vessel. With this end in view, safety vessels—the so-called sugar recuperators—are placed in the path of the steam in the triple-effect systems to catch entrained sugar.

The primitive anti-frothing arrangement of the early days.—Formerly vesicular carrying over was unknown, but they had to contend against the copious frothing which was generated in the vessels, owing to the imperfect working of the defecation processes. The safety vessel was then called the froth-breaker, and its rôle was much simpler. But it was very inefficient, and, above all, did not arrest the saccharine vesicles. Sugar in the boilers was then a common occurrence in sugar factories.

The Hodek.—Later on, as the methods of work improved, it was seen that, although they no longer had any froth, sugar was still carried over. It was then that an Austrian called *Hodek* invented the appliance which bears his name, and which paved the way for the *valentisseur* or break, almost exclusively employed in modern quadruple-effect systems, which does all that is necessary in connection with vesicular transference.

The froth-breaker was a closed wide column, still to be found in many an old or obsolete plant, which was intercalated between each vessel, and which contained in its interior a pipe of the same diameter as the steam pipe, and rising nearly up to the top of the column. The steam entered by the side, and was forced to ascend a little to find the orifice of the central tube, which it had to traverse before entering the next vessel. It will be seen that the steam impinging on the column might abandon the heavy particles such

as froth, but not the light vesicular particles which it carried in its train. Hodek sifted or sieved the steam. To do this he made it traverse a horizontal cylinder containing several pieces of wire gauze, on which it was bound to deposit its fine particles. Hodek's arrangement did its work well, but it created an obstacle to the passage of the steam, the speed of which was greatly slackened, thus lessening the turn-out of the evaporation plant itself.

Steam breaker.—After this the wire gauze in the Hodek was dispensed with, and the cylinder made wider and a little longer, and thus the steam breaker (*ralentisseur*), properly so-called, was obtained.

The theory of the *ralentisseur* had then to be established, because one could well explain that the steam in passing through a narrow into a wider tube would slacken its speed so that the heavy particles were precipitated. But on analysing this phenomenon mechanically it was seen that this fall could only be effected very imperfectly, if some other phenomena did not intervene to assist the mechanical action to stop the vesicles.

Horsin-Deon explains its action by the to-and-fro motion of the steam in the cylinder, as well as by the resulting changes of temperature, the dislocation of the vesicles, and the fall of the globules thus formed. A well-calculated and designed *ralentisseur* effectually arrests the vesicles of sugar in the steam.

Ralentisseurs are installed in different ways at the exit of the steam from the pans, according to the space at disposal. They are sometimes fixed horizontally, the steam entering at one end and issuing from the other, but the entry and exit may be either in the axis of the cylinder or on the side indifferently, although the axial position appears the preferable one, especially for the entrance of the steam. Sometimes the *ralentisseur* is vertical, fixed on the dome of the vessel, of which it looks like the prolongation. This is a very good arrangement. Sometimes, again, it is fixed vertically on the side, and resembles an ordinary *safety vessel*; but in this case the entrance and exit are both in the top of the cylinder, with a vertical partition between the two which divides it into two unequal portions. This kind of arrangement is not so preferable, as it causes the steam to make a long circuit at a sharp angle.

Many other arrangements have been designed. But the *ralentisseur*, in all its simplicity, is the best of safety vessels.

Konig's sugar recoverer.—All manufacturers, says the maker of this machine, know the considerable loss of sugar by carrying over in the vacuum pans and the triple-effect vaporating vessels. They also know what small practical results have been obtained up to now with the numerous appliances invented for recovering the sugar carried over, or for the prevention of the same. Fig. 48 gives an idea of the principal arrangements of Konig's recoverer. The steam from the juice passes over a striated surface, formed by

a large number of metallic sheets, the peculiar arrangement and sections of which force the vapour to follow a very sinuous and divided path. The particles of syrup carried over are deposited along the striæ, and reunite together in each sheet, flowing by channels formed by the sheets towards a circular pipe which extracts them from the machine. In the case of the large triple-effect vessels

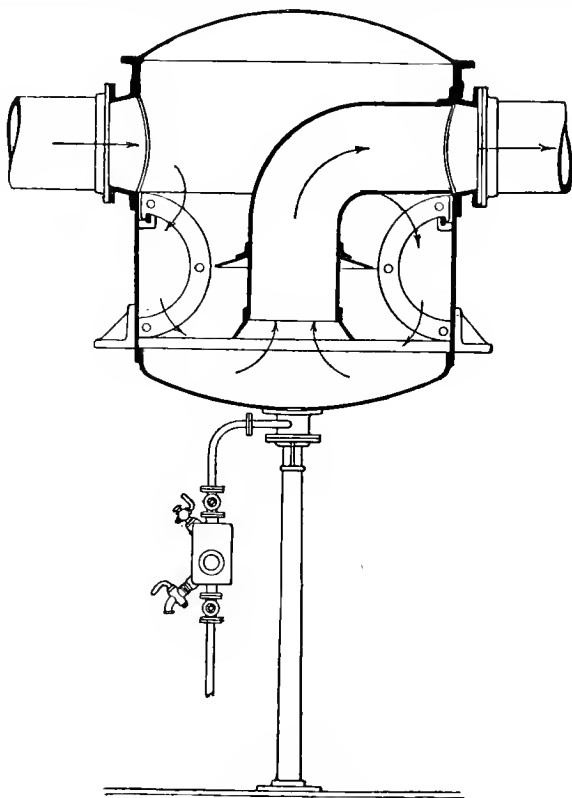


FIG. 48.—König's sugar recoverer (MAGUIN).

the area of the sheets is very considerable. The apparatus is made in two general types, as shown. One type of the recoverer is mounted on a triple-effect vessel. In the other pattern the recoverer is placed between the steam pipes, as shown in Fig. 48.

Hausbrand (in *Evaporating*, etc., pp. 117-160) treats the whole subject of the vesicular carrying over of liquids by their vapours and

similar phenomena in a very exhaustive manner, but the mathematical treatment of the subject is too intricate and involved for further discussion here.

WITKOWICZ'S RADIATING HEATER

The Russian engineer Witkowicz, in the trials in all the sugar factories managed or built by him with tubular appliances in the form of coils or lyres (Fig. 47), much used in Germany, Austria, and Russia, was struck with the great amount of heating surface left idle in all the vessels, and with the great benefit that would result from a circulation of the juice.

After making numerous experiments on mechanical motion and injection under pressure, he adopted the ingenious idea embodied in his radiating heater, which lends itself to numerous applications in reheating and carbonatation tanks, and in evaporation and vacuum pans.

Witkowicz's radiating heater consists of a steel-forged body in one piece, and completely closed. Two series of tubes in the form of an X (Fig. 36, p. 141) cross the body and impart to the juice, which passes over them, highly divided projections in an inverse direction, which produce an absolutely uniform circulation in the vessels where the radiating heater is fixed.

The ammoniacal vapours and the condensed water are removed with great facility and certainty. The radiating heater may be applied to existing evaporation vessels, or to tubular vacuum pans, so as to increase the production of the apparatus by more than 30 per cent.

The following are some of the applications made of this method in beet-sugar factories; it is quite adapted for heating by the juice steam, store tanks, juice, or syrup mixers, etc., in which there is no further need of mechanical motion.

THE RADIATING HEATER APPLIED TO EVAPORATION VESSELS

The application of radiating heaters to the *horizontal* vessels of the triple-effect system, whilst appreciably increasing the heating surface, also gives rise to an energetic circulation of the juice which did not exist previously, and increases their evaporative power by more than 30 per cent.

In *vertical* vessels several applications have been made of four radiating heaters placed under the lower tubular plate of the first vessel to increase its power and to allow of juice steam being raised from it for heating. An evaporation vessel or juice boiler with radiating heaters, placed in front of No. 1 vessel of the triple effect and heated by direct steam, furnishes heating steam and feeds No. 1

evaporating vessel. This appliance advantageously replaces the circulators. It may also serve as a No. 1 vessel for an ordinary quadruple effect, or as juice boiler before a quadruple effect.

CONDENSERS AND AIR-PUMPS

Their place and function in the triple effect.—The triple effect consists, as just seen, of three similar vessels—each provided with its sugar recoverer: No. 1 steam-heating No. 2 liquid, No. 2 steam-heating No. 3, whilst the steam from No. 3 passes to the *condenser* and the *air-pump*.

Two different methods of condensation may be adopted to produce the vacuum—

1. The production of a vacuum by the so-called *wet* system of condensation.
2. By the so-called *dry* system of condensation.

1. By this method, which is the one generally adopted, the course pursued is the same as that used for direct condensation steam engines. In this system the air-pump extracts the condensed steam and injected water along with any uncondensed gas. According to Hausbrand, the principle of opposite currents between steam and cooling water may be utilised in these condensers, but is not of great service. Wet condensers, he says, are generally arranged for parallel currents of steam and cooling water. Opposite currents, he says, may also be arranged in a wet condenser by admitting the steam below and exhausting the air above, so that the air being last in contact with cold water may be removed colder, which is an advantage in itself. However, he points out that the air in the pump cylinder, or even earlier, is in contact with the warm water, above which is steam of corresponding pressure. Thus any advantage of this construction, he contends, can hardly be recognised, for the air is ultimately mixed with the water, and very rapidly acquires its temperature, when the condition of things is the same as if air and water were exhausted by the same passage. The pressure in the wet air-pump is always dependent on the temperature of the water pumped out. A wet condenser with counter current (Fig. 49) consists of a vertical cylinder forming the continuation of the steam pipe from No. 3 vessel of the triple, No. 4 of the quadruple, or No. 5 of the quintuple system. Cold water is injected *underneath* by means of a rose (Fig. 46A), a cone, or a vertical pipe pierced with holes, and reaching to the top of the condenser (Fig. 49), or, finally, by any kind of arrangement which shoots the water in the form of finely divided spray in an inverse direction to the steam. A side tube C at the bottom connects the condenser with the air-pump. Now air-pumps, not having a variable volume, are sometimes drowned with condensed water when a large quantity is injected, and the air

can no longer be aspirated to such an extent that the vacuum is greatly affected. Attempts have therefore been made to separate the evacuation of the water from the removal of the air, and, to effect this, advantage has been taken of the water barometrical column.

Air-pump.—The air-pump is a large horizontal cylinder in which a piston is driven by a steam engine. Above the cylinder is a chamber with three compartments containing aspiration and pressure flap valves. The compartment in the middle communicates directly with the condenser, and its two faces, inclined laterally at 45° , are provided with aspiration valves.

The two extreme compartments communicate by apertures with the two extremities of the cylinder, and are furnished on their upper face, forming a roof, with pressure valves. On this chamber is fixed a tank with a tap for turning on the water.

At each stroke of the piston the aspiration which it produces causes the air of the condenser confined in the middle compartment, and the air which is in the condenser, to enter into the compartment communicating with it, and behind it, through the apertures in the cylinder. At the return stroke this air is pressed out through the valves into the upper tank, from which the water runs away and the air escapes into the atmosphere. The water which is left after this back movement of the piston overflows all the bad spaces, and when it starts again no trace of steam or air exists which might militate against the useful effect of the pump.

The great defect of too small pumps.—The pump ought to be sufficiently large to aspirate the water and the large amount of gas,

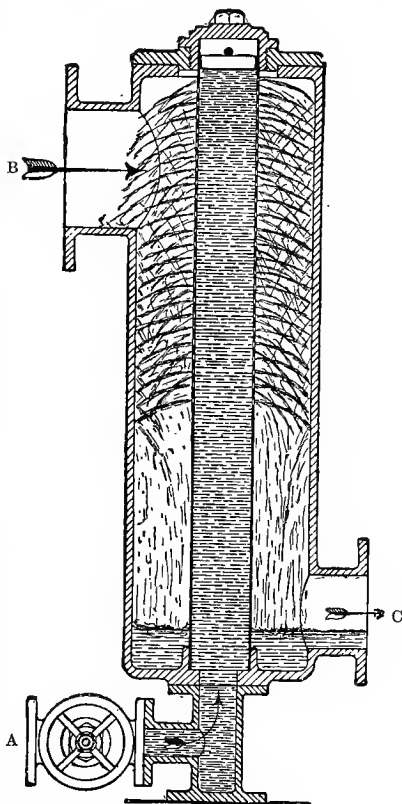


FIG. 49.—Reverse current condenser. A, Water injection valve; B, inlet for steam to be condensed; C, to air-pump.

coming not only from the juice but also from the condensation water itself, which is heated in contact with the steam. One of the great defects of the triple-effect system is the use of pumps that are too small.

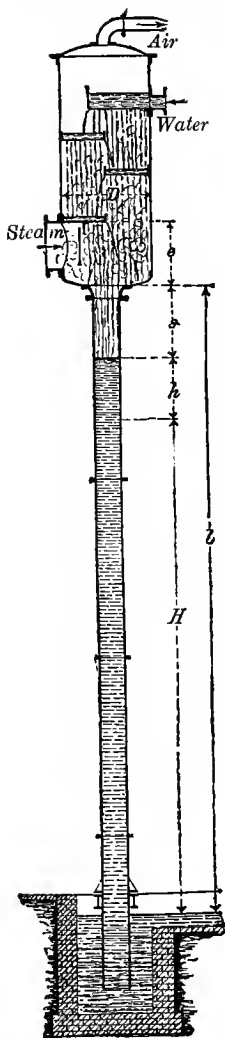


FIG. 50.—Parallel-current barometrical condenser.

2. *The fall pipe or barometrical condenser.*

—It is well known that an absolute vacuum corresponds to a column of water 33·8 feet high. If therefore a 33·8 feet pipe be placed, under the condenser, dipping into a tank of water, the water will rise in the pipe to a height corresponding with the vacuum, and will not rise any higher. Advantage has been taken of this fact, and the condenser has been placed 33·8 feet in the air, with a pipe running down to the ground floor of the factory, and dipping into a tank with a tap. When the cold water is injected into the condenser this water flows down into the pipe and escapes through the tank underneath. The requirements as to the condenser are, that it be large enough and that water can be injected into it in abundance and in a fine spray, so as to seize the steam immediately on its arrival. The barometric column with dry air-pump form of condenser is that which is mostly used in Britain, Austria, and Russia. It is also beginning to be adopted both in France and Germany. The object of this form of condenser is to relieve the air-pump of having to remove the condensed water as well as the air. The amount of water used in the condenser varies with the season of the year,—with, in fact, the temperature of the water to be used in condensation. The latter being colder in winter than in summer, less condensing water is required. Only, the shape of the condenser is slightly altered so that the air which it contains may be extracted from above. It still remains a vertical cylinder, but the steam enters on the side generally from below, and water is injected as a spray from above, whilst the air is aspirated through a pipe fixed on the side of the top of the cylinder (Fig. 50).

The water spray.—The mechanical division of the water is obtained by numerous artifices constituting a series of

cascares, which the air has to pierce before it gets to the top. During its impeded upward progress it is thus deprived of all the steam which it contained. Sometimes, however, the water and the steam enter at the top, and the air is extracted from the bottom of the cylinder. But generally the arrangement first described is preferred. In this connection, Hausbrand remarks that the principle of opposite or counter currents is almost always applied to this class, and with great effect, so that they are also called dry counter-current condensers.

Parallel- and counter-current condensers.—Nevertheless, in certain factories two condensers are placed side by side—the one with *parallel-current* of water and steam (as in Fig. 50), the other with *counter-current* (as in Fig. 49) (these are the terms applied to the two types of condensers), in such a way that the condensation is as perfect as possible and the air cooled to its maximum.

Dry air-pumps and wet air-pumps.—No water is extracted by the air-pump in this case, and that is why it is called the *dry air-pump*, in contrast with the other system called the *wet air-pump*.

These pumps ought therefore to be very perfect; bad spaces are avoided in them as far as practicable. They are real pneumatic machines. Thus the valves are frequently replaced by drawers or by cylinders, as in the distribution of the *Corliss* engines. Moreover, an arrangement is also in use which recalls Babinet's valve, which causes the bottom of the cylinder, when the piston is at the end of its course, to communicate momentarily with the other portion where the vacuum is at a maximum, so as to remove the air which may remain in the inevitable bad spaces.

Such pumps when well constructed often make a better vacuum than those of the first system. Moreover, they are smaller and less cumbersome than the others, which is a great advantage. Only—and that is the defect of the system as far as expense is concerned—they involve a force-pump to pump all the cold condensing water into a tank placed 36 feet up in the air, whilst the other system, being on the ground level, could draw its own water.

The pressure in the condenser.—The pressure in the condenser is the sum of the pressure of the air, gas, and steam. Warm water, which has previously been used for condensing, still contains some air, if it be only a small amount. In closed vessels partially filled with hot water, in which a considerable pressure is produced by artificial means, the water would still evolve steam of a pressure corresponding to its temperature, which would correspondingly increase the previously existing pressure. The function of the air-pumps, therefore, is to withdraw as quickly as may be the air and gas introduced by steam and juice, so that the pressure in the condenser may only consist of that due to the pressure of the steam, which depends on the temperature of the water. The pressure should be as low as possible, for as it decreases the boiling-point falls proportionately, and the useful

evaporating capacity of the heating surface increases in a similar ratio, as is pointed out in the Sugar Refining section, in the description of the pump of Howard's vacuum pan. There can be no question of exhausting by means of the air-pump the steam vapour formed from the juice, together with the air, since the volume of this vapour is so great that it cannot be withdrawn by pumps of rational dimensions. If it were tried to exhaust steam from the condenser by the air-pump, and thus to induce the formation of fresh steam from the water, which process would cool the warm water and so produce a greater vacuum, the air-pump would have to be of quite impossible dimensions.

Hausbrand (chapter xx. of his *Evaporating*, etc., p. 209) illustrates the above principles by the following example. (The reader is referred to this section of Hausbrand's treatise for much valuable information, for which no space can be found here.)

Example.—In order to condense 100 kilogrammes of steam, 3000 kilogrammes of water are required, which become heated from 15° to 35° C. To cool these 3000 kilogrammes of water 5° C., *i.e.* to 30°, it would be necessary to deprive them of 15,150 calories, *i.e.* to evaporate $\frac{15,150}{580} = 26.1$ kilogrammès. Now 1 kilogramme of steam at 30° to 35° C. has a volume on the average of 28,750 litres, thus 26.1 kilogrammes measure 750,375 litres. Such great volumes can naturally not be pumped out in a short time. It is therefore necessary to restrict the operation to removing the air alone from the apparatus as quickly as possible.

Quadruple effect, evaporation plant, with multiple-heating effects, Rillieux system.—Quadruple-effect systems only differ from the triple effect by having an additional vessel intercalated between the first and the last vessel. Horsin-Deon constructed a quintuple-effect plant comprising five similar vessels. Cail desired to make a nonuple effect, which would have likewise worked satisfactorily if the principles on which multiple-effect plant should be constructed had been understood at that time. Nothing, in fact, limits the number of multiple effects, unless it be the price of the plant, which beyond the quadruple or quintuple effect, with the complete Rillieux system, is too dear for the economy which it effects.

But with the usual complement of engines in a factory the triple effect is the only one possible, because, as soon as the quadruple effect, which consumes less steam than the triple effect, is started, there is an excess of waste steam, which is lost on the roof. It is therefore useless to get a fourth vessel when three suffice, unless the waste steam be utilised in the manner indicated by Rillieux.

The principle of the new Rillieux system is to regard each vessel of the quadruple effect as a steam boiler, capable, if of the right size, to furnish all the steam required to heat the next vessel, as well as for

the reheating of the juice, at whatever points may be necessary; whereas, therefore, we have hitherto studied only those multiple-effect evaporators in which the steam produced in No. 1 is used to heat No. 2, the same operation being repeated on No. 3 with the steam from No. 2 without interfering with the steam in any way whatever, either by taking from it or by supplementing it. But, in the new system, partially spent steam is frequently taken from Nos. 1, 2, etc., for reheating the juices. Steam is thus re-utilised, which has already

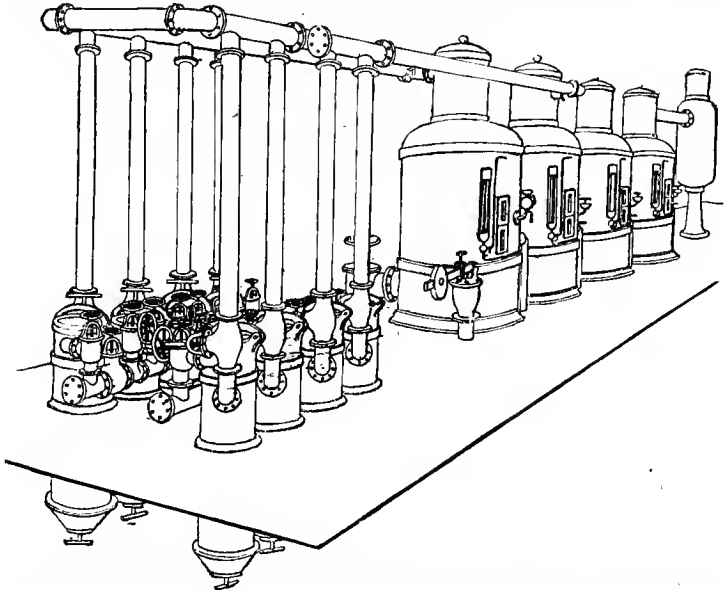


FIG. 51.—The quadruple-effect system of multiple-effect evaporation, and systematic utilisation of the steam from the vessels composing the series to reheat other juices.

removed several times its own weight of water in the multiple-effect plant.

The quantity of steam required and the rational utilisation thereof in the working of a quadruple-effect system, and general factory work.

No. 1 vessel.—Thus in the quadruple effect No. 1 yields steam at 106° to 108° C. ($222^{\circ}\cdot 8$ to $226^{\circ}\cdot 4$ F.). Then this steam is used to heat No. 2, and at the same time all the vessels in which it is necessary to bring the juice to 100° C. (212° F.), as in the second carbonatation, boiling the syrups, etc.

No. 2 vessel.—*The rational utilisation of its steam.*—No. 2 likewise furnishes steam at about 100° C. (212° F.). This steam, besides

heating No. 3, is used to heat all those vessels where it is necessary to bring the temperature of the juice to at least 90° C. (194° F.), as

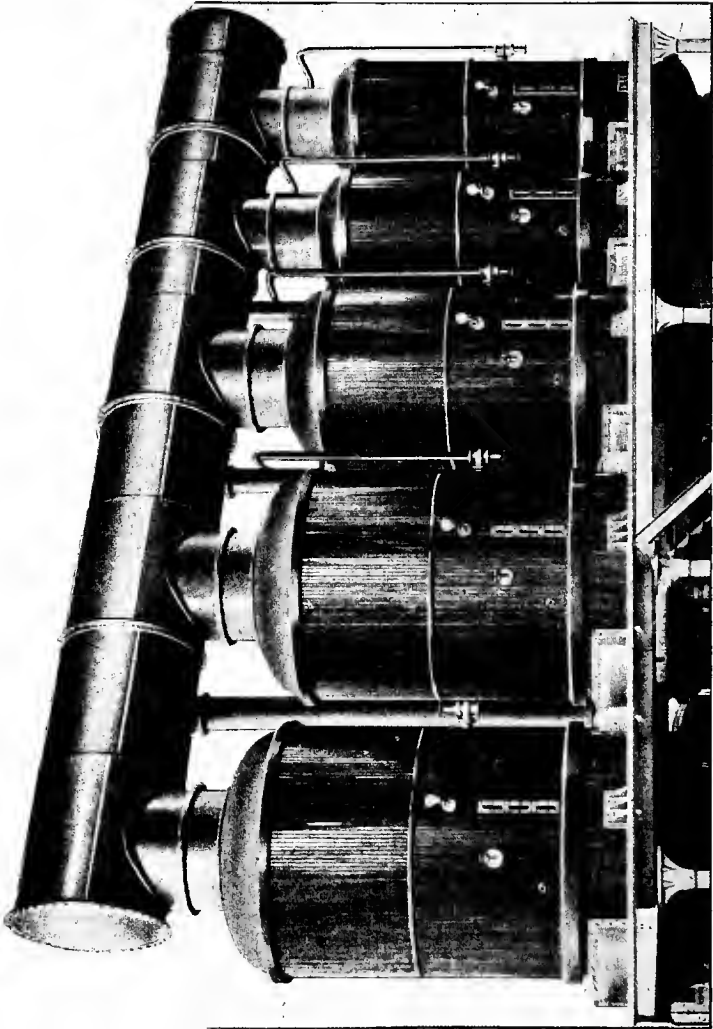


FIG. 52.—Quintuple-effect system.

in the first carbonatation, in the calorisors of the diffusion batteries, the coils in the top of the boiling pan, and so on.

The great strain upon the supply of steam in No. 1.—To do this it will be sufficient to calculate the quantity of heat which each of the vessels ought to furnish, and to impart to each of these vessels the heating surface by means of which they can evaporate that amount of water. Consequently, the No. 1 having to furnish steam for No. 2, independent of that which it will itself have to produce, will require to have greater heating surface than any of the others, and will generate all the steam necessary for all the heating required to be done in the factory beyond that required for the quadruple effect.

Assisted not only by waste steam from engines, but also by virgin steam from boilers.—It will therefore be necessary to furnish it, in addition to the exhaust steam, with the direct steam required for the rather considerable amount of work it has to do. It would, of course, be most advantageous to take all the steam required for the various purposes from the final vessel of the series, *i.e.* from No. 3 of the triple and No. 4 of the quadruple and No. 5 of the quintuple effect, but, as already pointed out, the temperature of the steam *falls* very decidedly from No. 1 to No. 4 or No. 5. The steam must therefore be taken from the vessel with that temperature which approaches most closely that of the work to be done.

Hausbrand gives the saving per 100 kilogrammes of steam withdrawn as follows:—

XLVI.

	Double Effect.	Triple Effect.	Quadruple Effect.
No. 1 . . .	47·5	31	22·5 kilos. of heating steam.
No. 2	62	45·0 „ „
No. 3	67·5 „ „

Water evaporated in each vessel when steam is taken from the individual vessels.—The production of the steam withdrawn may be regarded as quite distinct in all the vessels from the ordinary evaporation of the liquid, and it may be taken that these are separately introduced into No. 1:—

1. The water in the form of juice which is to be converted into steam in the various vessels, then to emerge partly as steam, partly as condensed water.

2. The liquor, which was originally mixed with this water but is now separate from it, and which, although it still contains the same quantity of solid matter as before, has that solid matter mixed with less water by the amount to be drawn off as steam for factory-heating purposes. The liquor is therefore more concentrated

than in the beginning. We can find the quantity of water to be evaporated in each vessel individually, and in the whole collectively, for the purpose of producing *steam for factory-heating purposes*. By subtracting this weight of water from the total weight of liquor we get the weight of liquor to be evaporated and condensed in the ordinary way.

With this system, therefore, there is no fear of wasting exhaust steam, since that yielded by the engines is insufficient even if the *quintuple* effect be used.

The economy thus effected in the use of steam.—What will be the steam economy realised under such circumstances? The reply is easy on the face of it, without calculations. No. 1 vessel yields steam from the juice for all the reheatings. To evaporate this juice it will require a weight of steam about equal to that which it produces. Application of heat is necessary, whether this steam be employed directly from the boiler in the coils or indirectly from the multiple-effect system. But in using it in the multiple effect a result is obtained which is not produced by sending the steam directly from the boiler through the coils, and this result is the evaporation of the juice. The economy of the system therefore consists in the evaporation of the juice with the steam, afterwards employed directly in the coils. This evaporation is therefore effected, so to speak, gratuitously, because it serves as the intermediary, almost without loss, to the use of direct steam. Moreover, the heating effected by the steam from No. 2 is produced by the steam coming from No. 1 in such a way that 1 lb. of steam evaporates 2 lbs. of juice before being drawn from No. 2 for reheating the juice in other stages of treatment.

The economy is thus double that obtained by taking steam from No. 1 for the purposes mentioned.

Weight of steam used per hectolitre of juice, or lbs. per 10 gallons.—The following is the distribution of the steam consumption in a sugar factory per hectolitre of juice (see also Table XLIII. p. 184):—

XLVII.

Operation.	Kilogrammes of Steam per hectolitre of Juice, or lbs. per 10 gallons.
1. Diffusion	8
2. First carbonatation	14
3. Second carbonatation	6
4. Evaporation (triple effect)	30
5. Heating of the syrup	1
6. Boiling (1st and 2nd)	8
7. Turbines and loss	10
	—
	77

If, therefore, the steam used in diffusion, carbonatation, heating of the syrup, say in all 29 kilogrammes, has at the outset evaporated 29 kilogrammes of water out of the 80 which must be eliminated, and the evaporation instead of 30 is now only 20, 10 kilogrammes of steam are therefore saved per hectolitre of juice, without going beyond the triple effect and heating by the No. 1 vessel; say 10 kilogrammes per 77, or an economy of about 13 per cent. If the triple effect be replaced by the quadruple effect, and if the steam used for reheating be drawn from No. 1 and No. 2 vessels, which is called *heating by the double and triple effect*, the steam consumption is reduced to 54 kilogrammes per hectolitre, say an economy of 30 per cent.

Its economy.—Thus with this system an economy of 30 per cent. may be effected in the steam actually used in sugar factories equipped with triple-effect plant—namely, instead of burning 120 kilogrammes of coal per metric ton of beetroots, only 84 kilogrammes are consumed.

The fuel consumption in Austrian factories, where Rillieux's system has been made a fine art, is reduced to a minimum.—That is what has made Rillieux's process such a success—a success which is being accentuated every day; for in Austria, where it has been equipped in altogether favourable conditions, by utilising all the multiple-effect heatings possible in reheating, boiling, filling, etc., they have reduced the fuel consumption to about 60 kilogrammes (132 lbs.) per ton of beetroot.

The coal bill, the chief item of expenditure besides the beet, reduced to a minimum.—The Rillieux process is therefore the process of the future, and every new sugar factory which adopts it in its entirety is assured of the minimum consumption of coal. Coal, after the beets, being the only serious expense in the factory, the great advantage of lowering the consumption thereof to the utmost limit will be readily appreciated.

The practical working of the quadruple effect.—The multiple-effect heating in conjunction with the quadruple effect is executed in the following way:—

1. The diffusion calorisors must be tubular, and have a heating surface at least double that of what they have when direct steam is used.

2. The limed diffusion juices pass into the reheaters, heated by the steam from the second vessel, and issue from these reheaters at about 80° C. (176° F.), and are then carbonatated in that condition without further reheating.

3. The juices going to the second carbonatation are heated to 100° C. in the reheaters, and go from thence to the second carbonatation pans.

4. The juices going to the quadruple effect are again brought to 100° C. (212° F.) in the reheaters by the steam from the first vessel.

5. The syrups are likewise heated by the first vessel, before filtration.

6. The vacuum strike pan is heated by steam coming half from the first vessel and half from the second, and its heating surface should be calculated accordingly.

Résumé.—It will be seen that the whole system consists in giving to each vessel of the quadruple effect the proportions suitable to the work it has to do, and passing all the liquids to be reheated into reheaters independent of the pan into which they are run, to be treated at the proper temperature. It is, in fact, necessary to give to the diffusion calorisors and the boiling pan heating surfaces sufficient for the steam from the multiple-effect system to bring the liquids to the desired temperature, all of which involves much calculation, and an installation does not succeed well unless the proportions are exactly prescribed and observed.

Reheaters.—The reheaters used in this installation are of two kinds:—

1. *Austrian reheaters.*—In Austria the reheaters are rectangular, and consist of vertical tubes packed between perforated plates, as in the vessels of the multiple-effect system, open from above to the air. The flow of the current of juice through them is rather slow, and the heating surface is also very great.

2. *French reheaters.*—In France closed reheaters are preferred, into which the pumps press so as to have a rapid current of juice. They may also be made of small dimensions, taking up but little room and working very regularly.

Either system works well, nevertheless,—the price of the plant alone differs; the installation is dearer in Austria than in France. But in each country each constructor has his predilections, and the engineer ought to humour his customers, so long as the final result does not suffer.

Extraction of the condensed water liquors from the individual vessels.—The condensed water in No. 1 comes from the waste steam of the engines which is used to heat that vessel, and from the direct steam from the engine boilers which is added as an auxiliary, if the engines do not furnish a sufficiency. This water is relatively pure, and is generally used to feed the boilers. The only impurity which it contains comes from the lubricants used for the steam cylinders. If *animal or vegetable* oils be used they are deposited on the tubes of the vessel with the condensed steam, and return to the boilers, where they may be dangerous on account of their corrosive action on the boilers. *Mineral* oils are therefore used, which are harmless. In No. 1 there is generally a pressure of $\frac{1}{2}$ atmosphere. The condensed water runs naturally away into the feed-tank of the boilers.

The ammoniacal vapour and ammoniacal pumps.—The condensed water from No. 2 and No. 3 vessels is charged with ammonia, because the boiling juice develops a great amount of ammoniacal

vapour. These ammoniacal liquors are not much esteemed for feeding the boilers, because *the steam which they give off, being charged with ammonia, would corrode the copper taps, the safety valves, etc.* However, by using a portion of it only, these mishaps are not to be feared, owing to the distillation of the ammonia into a large quantity of steam. But a vacuum exists in the second and third vessels, and to extract the water a pump is absolutely necessary; and, moreover, for the third vessel the pump should be well made, so as to cope with the high vacuum therein. These pumps, called *ammoniacal pumps*, should therefore be carefully constructed, because on their working depends the success of the system. Besides ammoniacal gases there are also present in the vessels the gases coming from the juice, and the leakage air which enters through the joints. It is therefore necessary to have for each vessel an air-pump to meet the wants of this double extraction.

Rillieux method of extracting the gases from the juice.—Now the constructors of the first Rillieux plant, in the making of the first sets of triple-effect plant, according to the plans brought from America by the German already mentioned, had neglected the removal of the air. The system therefore worked very badly, and it was by re-establishing the removal of the air, by a process other than the air-pump, that Rillieux was able, in 1882, to restore to the modern system the capacity of his old American plant. His system consisted in collecting the condensed water and the gases in a box in front of the ammoniacal pump, and in removing the air from this box by means of a gauged tap, which was furnished with a pipe going into a space, where a greater vacuum existed than in the box, say in the following vessel.

It was only after this was done that the European plant worked properly, and that they were able to construct quadruple and quintuple effects,—the attempts in this direction not having succeeded on account of the mistake made in not removing the air which encumbered the vessels.

Removal of ammoniacal gases.—In addition to air and the heavy gases, which accumulate in the bottom of the intertubular spaces, above the water, which enables them to be removed into the receptacles into which these gases with the water flow underneath, there is also in the vessel a certain amount of ammoniacal gas, which is *lighter* than steam. These light gases accumulate in the top, under the upper tubular plate. But ammonia in contact with copper attacks it, when a certain amount of air is present, as is the case in this instance. It has therefore to be removed as quickly as possible. That is done by making several apertures on the upper tubular plate, on which pipes are fixed, which coalesce into a single pipe, which emerges from the vessel on the outside (Fig. 55) and goes direct to the condenser. Owing to this arrangement the light gases are aspirated

to the condenser, and thus attack the tubes to a less extent than if they were allowed to remain in contact with them. A gauge tap, already mentioned, between the armatures and the calender is used to regulate the flow of the ammoniacal gases. In Witkowicz's radiating heater these ammoniacal gases (Figs. 36B and 55) are removed in a special way.

Mode of conveyance of steam from the engines and boilers to the multiple effect.—The triple effect further includes the steam pipes which feed No. 1 with the *exhaust steam from the engines*, all of which passes into a cylinder, the escape-steam cylinder, to be led from there through a large pipe into the intertubular part of No. 1 vessel. The auxiliary *direct steam from the boilers* is led into the escape cylinder, or into the vessel itself, by means of a balanced valve, which does not allow the steam to increase beyond measure the counter pressure in the escape cylinder, and thus stop the engines. According to Horsin-Deon, the valve invented by Rillieux does not really work well, except when its tongue is itself balanced so as to allow a large escape of steam with a slight upward pressure. The valve constructed by *Dulac* answers well for this purpose.

No. 1 is best fed with juice by the pump.—The juice is pumped into No. 1 vessel. Other and more complicated arrangements than the pump have been invented by different engineers, but the pump is the most correct means to ensure regular feeding.

No. 2 vessel aspirates the juice from No. 1, and so on.—No. 2 aspirates the juice directly from the bottom of No. 1, and No. 3 from the bottom of No. 2, whilst the syrup also is extracted from the last vessel by a pump. Formerly this extraction was made with a *montejus* working exactly like the sampling vessel, which has been already described. But the *montejus* deranges the work of the system at the moment of the vacuum. It is therefore nowadays almost everywhere condemned.

Mechanical filtration of the juice between No. 2 and No. 3 vessels, to avoid encrustation.—It is a good arrangement to filter the syrup between the last and the second last vessel. So as to avoid in this case the use of extraction pumps, closed mechanical filters are sometimes placed between the last two vessels in the path of the syrup intercalated on the feed-pipe. Phillippe's filter (Fig. 44, p. 173) is used for this purpose. This filtration has the effect of so far purifying the syrups as to greatly *reduce the encrustations* on the last vessel of the series.

Cleaning the triple-effect apparatus.—Finally, the different vessels of the multiple-effect system can be made to communicate with each other when it is necessary to empty them, which is only done when it is required to clean them or when the manufacture is finished. In America, Spenser used caustic soda to clean a "Yaryan," and found it to act effectively (see p. 311).

During the season 1899–1900 there were 255 sets of triple-effect plant in use in France, 93 sets of quadruple effect, and 16 sets of quintuple effect, whilst no less than 130 factories were using Rillieux's systematic method of reheating the juices by withdrawing the necessary steam from one or other of the vessels of the series forming the multiple effect.

Horizontal evaporation systems.—In all the foregoing the *vertical* triple and quadruple effects have been referred to. It was mentioned that Rillieux's original plant was *horizontal*, in the form of the tubular boilers of a locomotive. In Austria at the present time all multiple-effect plants are constructed with horizontal vessels. It will be useful to give a brief description, emphasising the points in which the horizontal differs from the vertical system.

The drawbacks of the original Rillieux plant.—Rillieux plant had several inconveniences. The height of the liquid was very great, on account of the *cylindrical* form of the vessel, and the space reserved for the steam above the juice was too small, because the tubes rose higher than half the circumference. Again, the tubes could not be dismantled, or only with difficulty, which was a great trouble in cleaning, which took more than one day on each occasion.

How remedied.—All these defects were remedied by first making the vessel *flat-bottomed*, which gave it the appearance of a trunk or a Watt's boiler. In this way the tubes spread parallel with this flat bottom occupy but a small height for an equal heating surface. Again, the juice only attaining to a small depth, the space for the steam was considerable. They therefore succeeded at the outset in avoiding the defects of the completely cylindrical vessel.

The method of fixing the tubes in the flat-bottomed horizontal Austrian vessels.—The manner of fixing the tubes is very ingenious. The tubular plates in the front and in the rear are pierced with holes of a wider diameter than the tubes. The latter pass through the plates, and on each is placed a vulcanised indiarubber washer. Finally, a cap taking eight tubes at a time presses with a single blow the eight washers against the tubular plates, and makes the joints tight. Eight tubes are therefore placed at a time. In cleaning, the cap, which is pressed and retained by a single bolt, is removed and the eight are at once freed. They are taken out to be cleaned, and another fresh set put in their place with their rubber washers and their cap. In a few hours, therefore, the whole tubular portion of the plant may be renewed, which is a great advantage for saving time in cleaning. Only, the rubber not being capable of being used twice over is so great an expense that these renewals are done as seldom as possible.

The streaming process.—The Yaryan.—The great obstacle to evaporation in all plant of this kind is the enormous mass of juice in circulation. Yet only the liquid which moistens the heated

partition is evaporated; all the rest only retards the departure of the steam, which occasions perceptible delay in the process, and consequently diminishes the output of the evaporation plant. If there were only a thin layer of juice on the tubes, just enough to moisten the surface, the pans would evaporate much more.

Attempts have been made to attain this object by causing the juice to stream over the surface of the tubes, leaving the interior empty for the escape of the steam. The first plant of this sort was invented by an American named *Lillie*, and caused but little commotion; whilst another American, *Yaryan*, adopting the same principle, made numerous claims in advertising his triple effect, and obtained brilliant results in evaporating water or pure liquors. But the highly encrustating juices seem to have rendered his system impracticable. However, opinion is divided, the successes balancing the failures, so that it is difficult to get at the truth. See under Cane Sugar (p. 311), where the Yaryan gave good results at Magnolia, and where the encrustation was effectually treated with caustic soda.

The matter being before the public, every one immediately tried to solve the problem by applying it to the existing plant. They placed on the tops of the vessels more or less well-conceived arrangements to distribute the juice in thin layers in the interior. But the matter is still in the experimental stage. However, it is possible the problem may be solved some day if encrustation does not form an insurmountable obstacle.

Automatic sampling of the syrup.—In order to ascertain exactly the nature of the virgin syrup which is used, during boiling, to strike-point, it has only to be analysed, an operation which is easily performed in the laboratories of sugar-works. But the difficulty is in taking regularly a fair average sample of the syrup during boiling. So as to get exact results, Guerin devised for this purpose the apparatus about to be described.

On the pipe leading from the filters to the boiling-down store tank, a branch pipe A, of 10 millimetres in diameter, is fixed, to which is adjusted a tap B, which is itself connected with the valve C. The valve is wrought by a counterpoised lever arm, to keep it usually closed. The exit of the valve is terminated by a reservoir R, of 1 to 2 litres capacity, ending in its lower part in a stopcock H.

The six-toothed wheel D lifts the arm of the valve F, every time it is set running, by the indices G and G', fixed on another wheel E. The index G' is shown as not working, on the illustration.

The indices are fixed on the sides of the toothed wheel E.

The wheel E is driven by an escarpment, the motion of which is due to an eccentric, as shown in the drawing. This escarpment may also be driven by a belt arrangement or by clock-work.

Manner in which the apparatus works.—The tap H of the reservoir is closed, and the tap A opened. The large wheel E is

driven at such a speed as to make one revolution every three minutes. If the index G be alone arranged for working, the small wheel D will make one revolution every 18 minutes; but as it only carries one stud which can work the arm of the lever F and the valve C, the valve only opens and shuts every 18 minutes. If the average

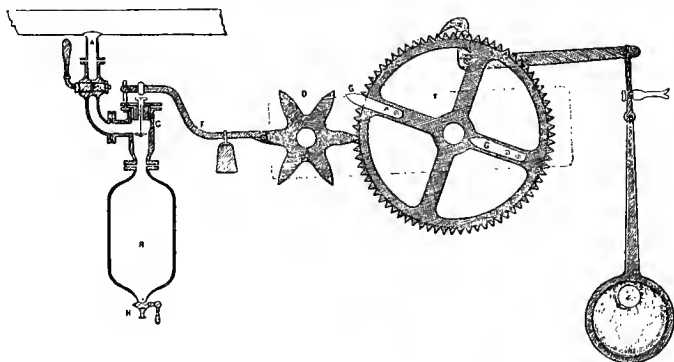


FIG. 53.—Guerin's automatic syrup sampler.

sample obtained in this way be not enough its volume may easily be doubled by placing the two indices G in the same position; the wheel D will then make complete a revolution every nine minutes, and the valve will lift itself twice as often as when only one index is at work. This apparatus eliminates all errors in taking the sample, whether they be accidental or intentional.

Another syrup-sampling device is described on p. 215.

CHAPTER VIII

THE EVAPORATION OF THE SYRUP TO CRYSTALLISING POINT IN VACUUM PANS—THE TREATMENT OF WEAK LIQUORS AND OF LIQUORS OF BAD QUALITY —TURBINES—MOLASSES

The following tables of the solubility of sugar at different temperatures, as well as the boiling-point of sugar at different pressures of the air, will be useful for reference.

XLVIII.—SOLUBILITY OF SUGAR AT DIFFERENT TEMPERATURES, WITH DENSITIES IN DEGREES BAUMÉ AND DEGREES TWADDELL, AND IN ACTUAL GRAVITY AT THE TEMPERATURE OF THE EXPERIMENT, AND AS BROUGHT TO 15° C. (FLOURENS).

Temperature ° C.	Sugar per cent.	Degrees Baumé.		Degrees Twaddell.		Actual Gravity.	
		At the Temperature of the Experiment.	At 15° C.	At the Temperature of the Experiment.	At 15° C.	At the Temperature of the Experiment.	At 15° C.
0	64·70	35·30	34·60	64·70	63·00	1·3235	1·3150
5	65·00	35·35	34·90	64·86	63·80	1·3243	1·3190
10	65·50	35·45	35·20	65·10	64·50	1·3255	1·3225
15	66·00	35·50	35·50	65·20	65·20	1·3260	1·3260
20	66·50	35·60	35·75	65·50	65·80	1·3275	1·3290
25	67·20	35·80	36·25	66·00	67·11	1·3300	1·3355
30	68·00	36·00	36·70	66·50	68·10	1·3325	1·3405
35	68·80	36·20	37·10	67·00	69·20	1·3350	1·3460
40	69·75	36·40	37·50	67·50	70·20	1·3375	1·3510
45	70·80	36·75	38·10	68·20	71·80	1·3410	1·3590
50	71·80	37·10	38·70	69·20	73·20	1·3460	1·3660
55	72·80	37·50	39·30	70·20	74·80	1·3510	1·3740
60	74·00	37·90	39·90	71·20	76·40	1·3560	1·3820
65	75·00	38·30	40·55	72·30	78·20	1·3615	1·3910
70	76·10	38·60	41·10	73·00	79·60	1·3650	1·3980
75	77·20	39·00	41·70	74·00	81·20	1·3700	1·4060
80	78·35	39·30	42·20	74·80	82·60	1·3740	1·4130
85	79·50	39·65	42·80	75·80	84·40	1·3790	1·4220
90	80·60	39·95	43·30	76·40	85·80	1·3820	1·4290
95	81·60	40·10	43·70	77·00	86·80	1·3850	1·4340
100	82·59	40·30	44·10	77·50	88·00	1·3875	1·4400

SOLUBILITY OF SUGAR IN WATER

237

XLIX.—SOLUBILITY OF SUGAR IN WATER AT DIFFERENT TEMPERATURES (DR. A. HERZFELD).

Tempera- ture ° C.	Sugar per cent.	100 parts of Water dissolve of Sugar.	Tempera- ture ° C.	Sugar per cent.	100 parts of Water dissolve of Sugar.
0	64·18	179·2	51	72·44	262·9
1	64·31	180·3	52	72·63	265·5
2	64·45	181·4	53	72·82	268·0
3	64·59	182·5	54	73·01	270·6
4	64·73	183·6	55	73·20	273·1
5	64·87	184·7	56	73·39	276·0
6	65·01	185·8	57	73·58	278·8
7	65·15	187·0	58	73·78	281·6
8	65·29	188·2	59	73·98	284·5
9	65·43	189·3	60	74·18	287·3
10	65·58	190·5	61	74·38	290·4
11	65·73	191·8	62	74·58	293·5
12	65·88	193·1	63	74·78	296·7
13	66·03	194·4	64	74·98	299·8
14	66·18	195·7	65	75·18	302·9
15	66·33	197·0	66	75·38	306·4
16	66·48	198·4	67	75·59	310·0
17	66·63	199·7	68	75·80	313·5
18	66·78	201·1	69	76·01	317·0
19	66·93	202·5	70	76·22	320·5
20	67·09	203·9	71	76·43	324·4
21	67·25	205·4	72	76·64	328·3
22	67·41	206·9	73	76·85	332·2
23	67·57	208·4	74	77·06	336·0
24	67·73	209·9	75	77·27	339·9
25	67·89	211·4	76	77·48	344·4
26	68·05	213·0	77	77·70	348·8
27	68·21	214·7	78	77·92	353·2
28	68·37	216·3	79	78·14	357·6
29	68·53	217·9	80	78·36	362·1
30	68·70	219·5	81	78·58	367·1
31	68·87	221·3	82	78·80	372·0
32	69·04	223·1	83	79·02	376·9
33	69·21	224·8	84	79·24	381·9
34	69·38	226·6	85	79·46	386·8
35	69·55	228·4	86	79·69	392·6
36	69·72	230·3	87	79·92	398·4
37	69·89	232·3	88	80·15	404·2
38	70·06	234·2	89	80·38	409·9
39	70·24	236·1	90	80·61	415·7
40	70·42	238·1	91	80·84	422·3
41	70·60	240·2	92	81·07	428·8
42	70·78	242·3	93	81·30	435·4
43	70·96	244·4	94	81·53	442·0
44	71·14	246·6	95	81·77	448·0
45	71·32	248·7	96	82·01	456·3
46	71·50	251·0	97	82·25	464·0
47	71·68	253·3	98	82·49	471·7
48	71·87	255·7	99	82·73	479·4
49	72·06	258·0	100	82·97	487·2
50	72·25	260·4			

Strike pans.—The syrup as it comes from the triple-effect evaporation plant has a density of 25° to 30° Baumé, sp. gr. 1·2 to 1·25, that is to say, during that operation 80 to 85 per cent. of the water which it contains is removed as steam.

Preliminary filtration.—It is now necessary to concentrate it to the crystallisation-point. At the outset it is filtered, because it is turbid. The methods of filtering the syrup are identical with those for the juice, whether through animal charcoal (Fig. 41, p. 160) or mechanical filters (Fig. 44, p. 173). Filtration through animal charcoal is, however, almost totally abandoned.

The filtered syrup is collected in a store tank, from which it is drawn by the sugar boiler.

VERTICAL VACUUM PANS.

The operation of boiling consists in evaporating and concentrating the syrup to crystallisation-point in an apparatus called a vacuum pan.

It is a pan provided with a steam coil, in which the evaporation process is finished in the vacuum produced by a condenser and an air-pump, as in the case of the triple effect.

L.—BOILING-POINT OF SUGAR IN A VACUUM PAN AT DIFFERENT PRESSURES OF THE AIR.

Height of Barometer in inches.	Boiling-point.		
	° Fahrenheit.	° Centigrade.	° Reaumur.
0·74	115	46·11	36·89
0·86	120	48·89	39·11
1·01	125	51·67	41·35
1·17	130	54·44	43·56
1·36	135	57·22	45·78
1·57	140	60·00	48·00
1·80	145	62·78	50·22
2·05	150	65·55	52·44
2·36	155	68·33	54·67
2·72	160	71·11	56·88
3·10	165	73·89	59·11
3·52	170	76·67	61·33
4·00	175	79·44	63·56

Description of the vacuum pan.—The vacuum pans are cylindrical vertical vessels (Fig. 54), surmounted by a dome, terminating in the lower part in a conical portion, truncated in a plane perpendicular with the axis, in the centre of which is a door of 20 to 30 inches, through which the boiled mass is discharged (Fig. 55). This door

is closed by contact against its armature, both door and armature being perfectly smooth, and sliding the one on the other horizontally round an eccentric axis in opening for discharging. There are generally three or four coils in the pan, touching the sides, as far as the door, and an additional coil in the centre. Each coil has its tap on the outside, but all branch off from one main pipe receiving

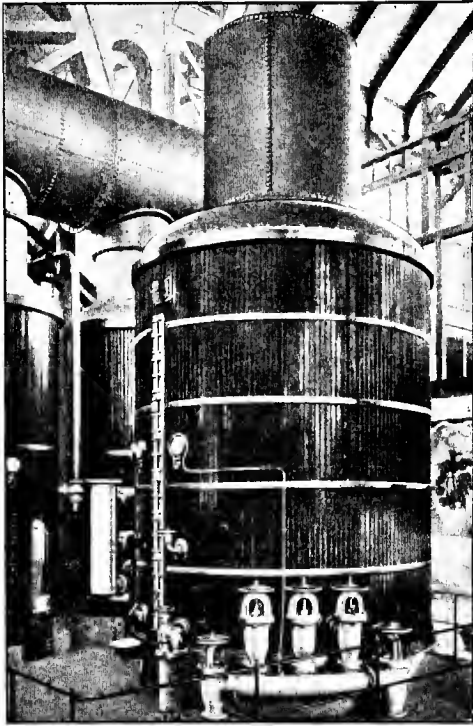


FIG. 54.—Vacuum pan (C&I).

its steam direct from the boilers. The cylindrical portion carries oblong lenses, arranged on a frame running up the whole height of the pan (Fig. 54). It has, like the multiple-effect pans, its *butter* tap, a second larger tap for breaking the vacuum by allowing the air to enter, so that the mass may be evacuated, and a pressure gauge indicating the vacuum in the interior. Finally, on the lower side, well within the reach of the boiler, is a *proof-stick*, to take a sample

of the mass, and to judge of the state of the boiling. The proof-stick is simply a long rod, 2 to $2\frac{1}{2}$ inches in diameter, dipping into the mass, and sliding by gentle friction in a bronze armature or sheath. On a point of the rod near its extremity is a depression. When the proof-stick is drawn towards the operator the depression comes *outside* the armature or sheath, the end of the proof-stick still corking up the opening. In the depression there is left a sample of the boiled mass, and from this sample, which is removed by the finger, the condition of the mass being boiled is ascertained. (See p. 357 *et seq.* and Fig. 79.)

Boiling to proof.—*String proof.*—Formerly boiling was done in open pans and over a naked fire, and the progress of the operation was judged by the appearance, between the finger and thumb, of a drop of liquid taken as a proof. If this drop, pressed between the index finger and thumb and the two rapidly drawn apart, formed a long, continuous, unbroken thread, it was said to have got to the stringing-point. Hence the term, boiling to string proof; if the thread broke, forming a longer or shorter hook, it was said to have got to the *light hook* or the *strong hook*. The latter was the sign of the finish of the boiling; the syrup then only contained 12 to 15 per cent. of water. This syrup was then run into tanks or moulds, which were left in a stove, and at the end of a certain time the whole crystallised.

With closed boiling pans, working *in vacuo*, the boiling is done by the grain.

Feeding the crystals.—It has been observed, in fact, that when several successive additions of lighter syrup are added to the boiling mass, small, almost imperceptible crystals are formed in it, and if the feeding with this syrup be continued the crystals grow, until after a certain time they become as large as a crystal of coarse salt. By feeding in this way the grain is nourished without forming any more additional crystals, and the skill of the boiler is to know how to obtain regular-shaped crystals, all of the same size. This is called *boiling in grain*. When the pan is quite full of this mass of grains, moistened by a scanty liquid called molasses, the *masse cuite* (boiled mass) has been obtained, and the process is pushed as far as possible by evaporating the last portions of water from the molasses, so that the mass only contains 5 to 6 per cent. of water.

Discharging the contents of the vacuum pan into shallow tanks.—When this occurs the operation is finished. The engine is stopped, the vacuum is broken, the discharge door is opened, and the mass falls into flat tanks, placed underneath, and arranged so that they may be easily emptied.

Boiling in grain.—Such is summarily the method of boiling in grain, which yields a crystalline mass directly, and which considerably abridges the time occupied by the old methods in open pans, whilst

it at the same time yields better and whiter products. During the season 1899-1900 there were 540 vacuum pans of enormous capacity in use in France for boiling in grain (*à cuire en graine dans le vile*).

HORIZONTAL VACUUM PANS

Rillieux economises steam in heating the vacuum pan.—It has been mentioned that in Rillieux's process boiling was effected by using the steam of the first and second vessel to evaporate the mass to strike point. In order to work in this way a large heating surface is required, and the strike pans now described are not at all adapted for that kind of work.

The kind of pan suitable for utilising the steam of the first and second vessel.—The pans used for this purpose are horizontal, of the same construction as the pans just described, only the flat bottom does not answer well for evacuation purposes.

The Lexa types of horizontal vacuum pans.—Austrian models.—Lexa, who made a special study of the construction of horizontal strike pans, has made two types of vacuum pans in Austria.

1. In his first model the flat bottom is replaced by three cones, each of which is provided with a steam coil to heat the mass in the cone, and a discharge door.

2. In the second type, which is much more elegant and practical, the flat bottom remains, but it consists of two movable doors, which slide horizontally under the pan, to the right and to the left, in such a manner as to leave the whole of the bottom wide open, and thus let the whole mass flow out rapidly. These horizontal pans are monumental in style, and are gradually replacing all the vertical pans formerly in existence in Austria. They are gradually being adopted in France. These pans constitute a real step in advance, both from the construction point of view and that of a more economical utilisation of the factory steam, and also because the boiled mass has a better appearance and the yield is higher.

Less injury from steam heat in horizontal vacuum pans than in vertical.—In fact, with direct steam in the coils (Fig. 55) the syrup is burned, the steam is too hot, therefore the boiled masses are very dark in colour. With horizontal boiling, on the contrary, the steam is colder, does not darken the syrup at all, and the mass is white and rich.

Rillieux's original vacuum pan a horizontal one.—Moreover, the first vacuum pans made by Rillieux were horizontal and heated by No. 1 steam, and the reports made in 1845 relate that the boiled masses were crystalline and blonde—a result which could not be obtained with Howard's vacuum pan.

The radiating heater applied to the "firsts" vacuum pan.—All the old types of "firsts" vacuum pans had insufficient heating

surface, but with the radiating heater it is claimed that the heating surface may be doubled or even trebled whilst still retaining one or two coils in the bottom of the vessel to form the grain. Fig. 55 shows two radiating heaters, installed in any ordinary vacuum pan, replacing a removed coil. The radiating heaters may be heated by high-pressure steam or juice steam, and their adoption gives great elasticity to the work. The circulation of the boiled mass

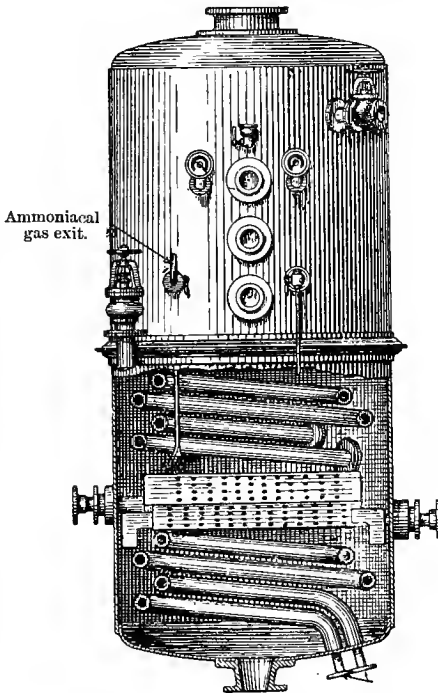


FIG. 55.—Radiating heater applied to a vacuum pan replacing a removed coil (MAGUIN). (See Fig. 36B.)

becomes very energetic, by the agitation which is imparted to it in two different directions in the tubes of the radiating heater. Crystallisation is facilitated by the motion imparted to the boiled mass. Exhaustion is better, and the duration of the boiling is very much reduced.

The radiating heater applied to the "seconds" vacuum pans.—In working the "seconds" the vacuum pans have a still smaller heating surface than those intended for "firsts," and the radiating heater brings a great improvement to bear on their interior arrangement

and in the working of the product. Numerous improvements have been made in Germany and in Russia on existing "seconds" vacuum pans, generally tubular, with small coils and of horizontal shape. A "seconds" jet vacuum pan with motion may be fitted with the radiating heater, so as to realise all the advantages of Hach and Laucke's mixers without their numerous drawbacks, the most serious of which is that when it is desired to continue the boiling in these vessels there is insufficient and badly arranged heating surface.

The Bocquin-Lepchinsky cooler.—A system, only a few years old, and which is now much used, is Bocquin-Lepchinsky's cooler. It is a large tank with a round bottom, in the horizontal axis of which an agitator revolves. A current of cold water circulates in an outer jacket. This tank has the capacity of a vacuum strike pan, and, during the whole of the time occupied in cooling, the agitator is kept in motion. The result is that, in spite of the cooling, the crystals remain detached from each other, and are still fed; whilst in ordinary tanks, or the Schutzenback's, the boiled mass solidifies, thus preventing regular crystallisation. The method, thanks to this motion, gives therefore a greater yield and larger crystals than cooling in tanks.

Crystallisation in motion.—This method is called crystallisation in motion, and its principle is applied to all the products of the sugar factory, whilst Bocquin-Lepchinsky only applied it to the "firsts" *masse cuite*.

The principle on which the phenomenon depends.—If crystallisable molasses, to which a piece of sugar crystal has been added, be stirred, the crystal increases, and a result is obtained after a few hours' mechanical working which could only be got after the expiry of several months by the old process. This is a step in advance which is occupying attention, but which is not known generally enough to enter completely into the domain of actual practice. It is only in the experimental stage, but these experiments have been so successful that there is every reason to hope for a great future for crystallisation in motion. In 1899-1900 there were, in fact, 191 factories in France working with *open crystallisation in motion*, and 20 employing the same principles in *closed vessels*. (See pp. 255, 260-264, and 279.)

Storing and cooling vats.—The boiled mass, according to what has been said, is simply syrup, of which a large portion has separated out in the form of crystals. The molasses which moisten these crystals contain, besides sugar, all the impurities of the defecated juice. If the boiled mass be allowed to cool, the sugar which it contains in solution still continues to crystallise round the existing crystals by molecular attraction. But the crystals are not so pure and are less brilliant than those contained in the hot mass. Nevertheless, it is advantageous, from the point of view of immediate

production, to allow the mass to cool, if regard be not had to an extraordinary beauty in the crystals. Certain plans have been made to effect this cooling under the best possible conditions. The simplest way is to place large shallow tanks underneath the vacuum pan, and to let the boiling mass drop into them.

The utility of the Schutzenback small tip-tilting tank waggons.—Another method, due to Schutzenback, is often used. The mass is run into a series of small truncated tanks, which are charged with only 2 cwts. These are stored in a cool room until completely cold. This involves much handling, but they present great advantages, by facilitating the turbine process, as will be described in the sequel. They are handled by means of small trucks, consisting of two large wheels, between which each tank can be caught by lugs fixed on it for the purpose. They are thus easily transported, and can be pivoted on themselves by completely tilting them, when it is desired to empty them, without assistance and without any great exertion.

ANALYSES OF SYRUPS AND "MASSES CUITES"

Sampling.—In the case of *syrup* the fair average sample is obtained by means of Guerin's syrup sampler (Fig. 53), as described on p. 235. The sample, on reaching the laboratory, is well triturated, so as to ensure thorough admixture.

Precautions in sampling the "masses cuites."—To get a fair average sample of the *masse cuite* requires careful working. The sample must be taken from the actual flow of the *masse cuite*, because when it is deposited in the tanks in the fill-house it is impossible to take a fair sample, as the crystals commence to deposit immediately on the bottom of the vats. Some *masses cuites* have, moreover, a tendency to deposit very fine crystals; it is therefore advisable to triturate the sample taken, so as to get a uniform mass. The "seconds" *masse cuite* often produces, on stirring, an abundant granular crystallisation, and the whole mass assumes a crystalline appearance, like that of honey. These small matters require attention, else a fair average sample for analysis will not be obtained.

Analysis.—In the case of *syrup* either 50 c.c. or 50 grammes are taken for analysis, according to whether the result is to be expressed per litre or per 100 kilogrammes. In dealing with *masse cuite* 50 grammes are invariably taken, dissolved in distilled water, and after cooling made up to a volume of 250 c.c. The liquid thus obtained, being a 20 per cent. solution, may be used for all the estimations that have to be made. Moreover, the density of the liquid at 15° C. will show the apparent percentage of dissolved matter just as well as the density of the *masse cuite* itself.

Sugar estimation.—A solution of basic acetate of lead is added

to 100 c.c. so as to make up the bulk to 110 c.c. Instead of multiplying the degrees read off by the normal weights, they are multiplied by the following coefficients—0.896 for the French saccharimeter, and 1.433 for the German one.

Ash.—For the ash determination, 5 to 10 c.c., corresponding to 1 or 2 grammes of substance respectively, are taken, 2 drops of sulphuric acid are added, and the whole evaporated on the water bath. The dry black mass is inserted in a muffle furnace, and the incineration proceeded with in the ordinary way.

Purity and saline coefficient.—The calculation of these from the data obtained presents no great difficulty. To get the real actual purity an exact moisture determination must be made, working on 10 c.c. of liquid. This determination is made by Josse's striated filter-paper method, rolled on a bobbin having the same dimensions as the capsule. The paper absorbs the liquid, and spreads it over a considerable surface. It is dried at 105° C. until of constant weight.

The actual dissolved matter may be approximately determined from the apparent percentage of dissolved substances. For that purpose the amount of non-saccharine is determined, which is multiplied by the coefficient 0.93 and added to the sugar. The result is very near the actual amount of substances dissolved. By subtracting it from 100 the remainder gives the water in the sample tested.

Alkalinity.—The alkalinity is determined on 50 c.c., using sensitive litmus paper as indicator.

Total lime.—The total lime is determined by means of the standard soap (hardness) test on 20 c.c. diluted with its own bulk of distilled water. The degrees are multiplied by 0.0057 so as to get the percentage of CaO, the circumstances being quite identical with the analysis of a water; the proportion 20 c.c. = 4 grammes per 100 = 40 per 1000. Sidersky's burette (Fig. 38, p. 148, and context) is so graduated as to render any calculation unnecessary.

Determination of the quantity of crystals in a "firsts" "masse cuite."—The remainder of the sample taken for analysis is placed in a funnel, the stem of which has been loosely packed with glass wool or asbestos fibre. A previously tared platinum capsule is placed underneath, to collect the raw syrup adhering to the crystals. The capsule is weighed in order to get the weight of syrup; a drop of sulphuric acid is added, and the syrup incinerated. If C be the weight of the ash of the raw syrup, and C¹ the weight of the ash of the *masse cuite*, the quantity of green syrup per cent. of *masse cuite* will be $\frac{100 C^1}{C}$, and the percentage of crystals = $100 - \frac{100}{C}$.

Stocktaking and balancing the work from the measurement of the "masse cuite," and its analysis.—The density of the *masse cuite* and its saccharimetrical analysis having both been determined,—if its volume be also known, then the total quantity of sugar produced is also

known. This amount of sugar ought to be identical with that contained in the beets treated, which have been weighed and analysed. The *masse cuite* contains the whole of the sugar of the beets minus losses. Its analysis therefore is a safe guide in ascertaining the good or bad working of the factory.

The *masse cuite* cannot be weighed except when Schutzenback's tanks are used. This is rather unfortunate for stocktaking purposes, because, if the weight and percentage strength of the sugar were known, then the whole of the working data would be at hand, so as to balance the working accounts of the factory. The large cooling vats cannot be weighed; their measurement and the density of their contents are taken, and the total weight calculated from the data thus obtained. This weight is far from accurate. But in actual sugar-works practice it is continuously determined; in the end the errors correct themselves, and a sufficiently exact statement of each run is obtained. The analysis itself is not free from difficulty. It is difficult to obtain a fair average sample, and this failure gives rise to numerous errors.

But, in any case, the analytical results of the *masse cuite* are those by which not only the amount of sugar extracted from the beet is determined, but also the quality of sugar is thereby ascertained, and the good or bad working of the factory.

Saline coefficient.—When dealing with good beets the boiling to strike-point results are good, and the ratio of the sugar to the salts contained in the mass is high. This ratio is called the *saline coefficient*, and varies about 20. Above that figure the results are good; below, down to 16, middling; less than 16 denotes bad beets highly charged with saline matter. The *saline coefficient* is therefore a sure guide to warn the manufacturer of the necessity of trying to improve the quality of the *masses cuites*. If this be not done, a poor yield of sugar and much molasses will result.

It is, however, impossible to determine exactly the volume of the "firsts" *masse cuite* generally run into large, flat, shallow tanks of great width. A few small fractions of an inch, more or less, produce a great difference in the cubic content. But not only so, the data to be produced from the volume of the "firsts" *masse cuite*, however exactly ascertained, are completely spoiled by the return to it of the turbine drainage liquors.

It has been suggested to put these figures to one side, and only to take as a basis the turbinated sugar and the "seconds" *masse cuite*. It is plain that the sum of the sugar contained in the turbinated products and in the drainages is equal to the sugar comprising the "firsts" *masse cuite*, without any addition of turbine drainage. The turbinated sugar is generally weighed, and the "seconds" *masse cuite* is run into vessels deep enough to obviate any error in gauging them. All that has to be done, therefore, is to get a fair average sample of the above

products, polarise the turbined sugar, and determine the density of the molasses and the "seconds" *masse cuite*, and all the data are to hand for determining the quantity of sugar contained in the "firsts" *masse cuite*.

DIFFICULTIES IN BOILING, AND JUICES IMPOSSIBLE TO BOIL

According to locality and circumstances, there are sometimes difficulties in boiling syrups.

These may be classified as due to the following causes:—

1. Presence of gummy substances in the juice.
2. Imperfect lining.
3. Fermentation of the juice.
4. Excessive alkalinity of the syrups.

In the first two cases boiling is slow and difficult, but it is always possible to form the grain. In the two latter cases it is impossible to do so without having recourse to roundabout methods.

1. The first case is due either to the beetroots being spoilt by too long storage in the silo—a mishap which is apt to occur towards the end of the manufacture—or in consequence of excessive heat prevailing at the commencement of the manufacture, in certain countries, preventing any preservation of the beetroot (South of France, and Spain). Under the influence of the ferments which are generated in the beetroot a portion of the sugar is transformed into glucose. Moreover, the products which are generated during this change are exceedingly viscous.

Soon after the boiling has begun the vacuum rises to 20 to 21 inches, the syrups scorch, and the air bells or bubbles have great difficulty in effecting a passage for themselves. In order to form the grain it is necessary to lower the vacuum as far as possible, and for this purpose to withdraw almost all the injection water, and to give very small and often-repeated charges, *i.e.* feed by small gushes. After a certain time the vacuum gradually lowers, small crystals appear on the glass, and the operators, in examining them, feel that the liquid which surrounds them is *charged* with gummy substances. The nutrition of the grains is very difficult, on account of the viscosity of the syrup, which holds the crystals like the meshes of a net. The boiling must be done very slowly and regularly. The syrup assumes a deep coloration, its colour becoming accentuated to a black-brown. As to pressing the boiled mass, it can only be done very badly, and never but very incompletely.

Turbining ought to be done very rapidly, for, if the boiled mass begins to cool, the sugars purge with difficulty, and in that case it is necessary to further refine, which results in a further loss of white sugar. In order to obviate this inconvenience, it is necessary, in hot countries, either not to take delivery of beetroots in advance, or to

make a judicious mixture of fresh and spoiled beetroots, so as to diminish the bad effects.

2. A difficulty in boiling, owing to a deficiency of lime in the first carbonatation, is immediately manifested. The syrup tanks are covered with an abundant white scum. In the boiling vessel the syrups froth enormously, with a tendency to prime.

A jet of direct steam can alone be used under such conditions; the evaporation is therefore very slow. The froth may be partly suppressed, either by introducing grease, oil, or melted tallow into the vessel, or a little dilute hydrochloric acid, which instantly suppresses the froth. It is advisable to add it to the syrup tanks. As soon as the froth has been suppressed the steam may be increased, and evaporation is then effected more rapidly; the grain is easily produced, and the boiling process is pursued as under normal conditions. One must expect abundant frothing in the boiling of low-quality products, and also on the filling vessels, which is easily suppressed, as will be seen further on.

3. The third case is due to the fermentation of the juice after a temporary stoppage of the factory (breakdown of the machinery, muddy diffusion water, etc.), or the starting of a pulping machine.

In the boiling vessel the steam does not take hold upon these fermented juices, the manometer rises to 4 or 5 atmospheres, the vacuum rises to 25 or 26 inches, the juice remains cold, and a thick scum rests on the surface of the liquid.

In order to remedy this inconvenience, the only practical method to use is to pour into the boiling vessel 1 litre of a 5 per cent. solution of carbolic acid. The effect produced is immediate. The scum disappears, the vacuum falls down again to 17 to 18 inches, and the manometer makes a sudden backward movement. The liquid enters into ebullition, and the boiling, which was immobile and impossible, is as easily condensed as in an ordinary boiling. It is well to add a little carbolic acid to the syrup tanks, so as to anticipate the work.

4. If the difficulty be due to an excess of lime, the analysis of the syrup will at once detect it. As in the preceding case, the steam does not take hold on the juice, the manometer remains at 4 or 5 atmospheres, the vacuum at 26 to 27 inches, the syrup remains cold, but no surface froth is produced.

In such a case, hydrochloric acid must be used to saturate the excess of lime (the acid used is the commercial acid used for washing the triple effect). The operation is commenced by running 1 to 2 litres of the acid into the boiling vessel, stopping when the manometer is seen to redescend. Ebullition soon takes place. If it be seen that the effect is satisfactory it is useless to add more acid, as the result would be to wash the vessel, the juice would become black and be contaminated with particles of iron or scoriæ, which

would again contaminate the boiled mass and the sugar. It would be advisable, on the other hand, to add some to the syrup tanks. This would greatly facilitate working.

In spite of these precautions boiling is slow, and concretion difficult—lasting two hours, and very imperfect. In these conditions it is much preferable to make small boilings so as to gain time, because a boiling to the full capacity of the apparatus would require 17 to 18 hours.

Difficulty in boiling low-quality products.—To remedy this inconvenience, it is necessary to add to each boiling 1 litre of carbolic acid in 1 per cent. solution.

TURBINES

The magnitude of the centrifugal force in turbines.—In the case of a point revolving in a circle, of which the radius = R and the time of revolution = T, the magnitude of the centrifugal force is $\frac{4T^2R}{T^2}$.

Supposing R = 2 feet, and that the particle makes 10 revolutions per second, the magnitude of the force is therefore $4 \times \left(\frac{22}{7}\right)^2 \times 2 \times 100$. This quantity, when calculated out, gives 7884. But the force of gravity is 32; therefore the ratio of the centrifugal force to gravity is $\frac{7884}{32} = 246$. The centrifugal force is therefore 246 times greater than gravity. Each particle of water, therefore, between the crystals of sugar is expelled by a force 246 times greater than its own weight.

The great advantage presented by centrifugal force for separating the liquid molasses from the crystalline sugar lies in the fact that each atom of water is expelled by a force acting on itself directly, and not transmitted to it by the pressure of and through the intermediary of the surrounding particles.

Suppose, for instance, it were attempted to express the molasses from the sugar by hydraulic pressure, in the same way as oil is expressed from seeds, the particles of sugar on the outside of the cake would be crushed, but the pressure would be but very imperfectly transmitted to the interior. As a matter of fact, unless the crystals were so compressed and crushed together as to annihilate all the intervening interstices, the water would not be removed from the bulk of the mass. Such a result, it is needless to state, could not be obtained by pressure without crushing and destroying the beauty of the crystals beyond all recognition. On the other hand, when centrifugal force comes into play its action is altogether different: the crystals, though as tightly and closely packed together as possible, are quite uninjured; their crystalline symmetry remains intact, whilst at the same time numerous interstices intervene between the crystals,

and out of these the water is ejected by the centrifugal impetus of its own inertia.

LI.—THE TURBINE, ETC., EQUIPMENT OF FRENCH BEET FACTORIES,
1884-1900.

Season.	No. of Turbines.		No. of Factories possessing Weinrich Turbines or other Patent Process.	No. of Sets of Weinrich Turbines or other Patent Process.	No. of Factories with Koer- ting's Injectors or other Arrangements for produc- ing a Steam Spray.	No. of Koerting or other Injectors.
	Ordinary, Uncovered.	Ordinary, fitted with Covers.				
1884-85 . . .	861	1013	35	96	46	66
1885-86 . . .	694	1036	34	105	45	68
1886-87 . . .	661	1017	34	98	43	80
1887-88 . . .	670	1005	24	96	37	56
1888-89 . . .	721	1009	28	80	36	61
1889-90 . . .	762	992	18	80	33	62
1890-91 . . .	876	1013	16	73	36	63
1891-92 . . .	858	1026	16	82	34	60
1892-93 . . .	911	1079	13	73	31	45
1893-94 . . .	913	1132	11	49	26	49
1894-95 . . .	981	1164	10	62	20	30
1895-96 . . .	961	1179	9	51	25	38
1896-97 . . .	1073	1093	14	63	26	47
1897-98 . . .	1113	976	15	58	29	39
1898-99 . . .	1165	1045	22	72	24	46
1899-1900 . .	1217	1162	21	84	25	54

THE TURBINING OF THE BOILED MASS OR "MASSE CUITE"

The history of the turbine.—The idea of applying the principle of the centrifugal machine—called a turbine by the French—would appear to have been suggested by the turbines previously existing in establishments for the bleaching, etc., of textile fabrics. Moisture had been expelled from fabrics by centrifugal machines, or turbines, long before any one ever thought of using them in sugar-works. It is not known exactly when or in what country they were first used in sugar factories. In any case, in the year 1850 no less than thirty-six patents were taken out in Great Britain alone for the employment of new and improved forms of turbines for use in sugar factories.

The turbine has been aptly described as a wire gauze cup, strongly fortified with wire and with bars, mounted on a vertical pivot, and capable of revolving at as high a velocity as 1500 revolutions a minute. The turbine, in fact, consists of a vertical cylindrical open-

topped drum (with a vertical axis), 2 to $2\frac{1}{2}$ feet in diameter, the side of which consists of wire gauze strengthened in the manner indicated. The bottom is closed, and a conical portion surrounds the spindle or axis of rotation. The whole is enclosed in a cylindrical cast-iron case to match, having in its bottom an arrangement on which the axis or spindle rests.

Method and principles of working.—It may be driven from above by friction cones, or from underneath the drum by belting.

Speed.—In beet-sugar factories the speed is generally 400 to 600 revolutions a minute. Whilst revolving at this speed, if a certain weight of *masse cuite* be run into it—say 1 cwt.—the whole mass is projected against the side of the drum by centrifugal force. As the side consists of tight wire gauze the molasses pass through, whilst the crystals remain behind in the drum. By cleansing the crystals with water, pure syrup, or steam, or even by a blast of moist air, the final portions of the molasses which soiled them are eliminated, and white granulated sugar is obtained.

Types of turbines.—Turbines, as just mentioned, are of two types—

1. The French turbines, driven from above.
2. The Austrian turbines, driven from underneath (Fesca's type).

French type of turbine.—The French turbine, the axis of which is held by the two ends, is more stable and does more work. But the driving of it from above involves a cumbersome arcade, supported on the framework of the machine.

Austrian type of turbine.—The turbine driven from underneath allows the drum, which is also called the basket, to be got at in any part of its surface, which is a great advantage in filling. But the axis is held in two near points—(1) at the bottom, and (2) near the centre—in such a way that the latter is a false door. The result is that a nutation motion is produced on starting, which Fesca has corrected by a special regulator in the cone present in the centre of every turbine to regulate the spreading out of the *masse cuite*.

The French firms who have built this latter kind of turbine have partially corrected this inherent defect of the machine itself by placing the second point of attachment of the axis at the centre of the cone itself, in such a way that the centre of gravity of the turbine is between the two fixed points. They thus dispense with Fesca's regulator.

Nevertheless, the excellent qualities of the French turbine cause it to be preferred in spite of its defects, and the second is only utilised when there is no other resource, particularly in the special type of turbines for refined sugar, such as the Fremaux turbine, which necessitates the basket being quite open. The turbine shown in Fig. 56 may be made to do the work of two; whilst one drum is being discharged and refilled, the other is being rotated.

Weston's turbine.—There is a third kind of turbine, of American origin, used especially in cane-sugar factories—Weston's turbine. The basket is hung at the lower end of a vertical axis, which is only held by a single point above at the other end of the axis at the centre of the driving pulley, in such a way that the belt pulls on the point of suspension without interfering with the vertical position of the axis. These turbines have therefore been constructed so as to be

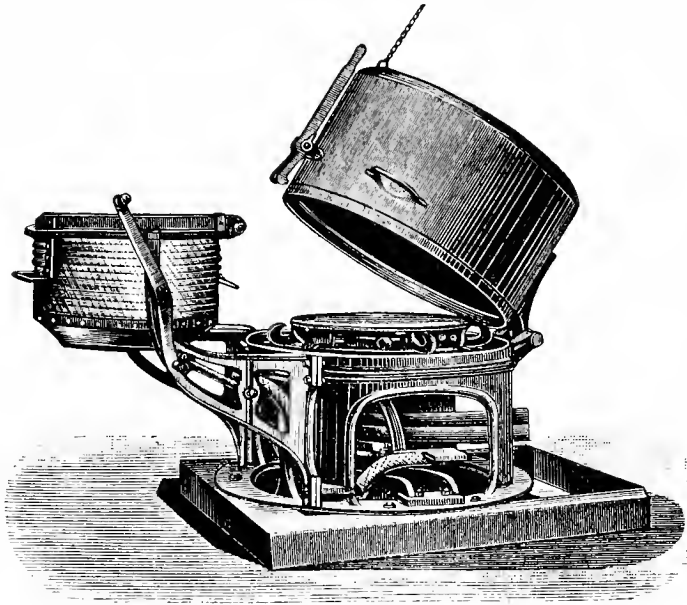


FIG. 56.—Turbine driven from underneath (HANBOLD).

emptied from below, the central cone being capable of being displaced vertically, leaving a hole wide open through which the sugar is pushed, without touching it by hand, by means of a small shovel. The sugar thus escapes being soiled by the dirty hands of the not very clean negroes.

French form of turbines discharging underneath.—The discharging of turbines from below, having some advantages, has also been effected in the case of the French turbines, by leaving an opening in the bottom of the drum, which closes with a sliding door, which corresponds with a funnel in the cast-iron cover in which every turbine is enclosed, and in which the turbinated molasses collect.

A great field open for continuous turbines.—A continuous turbine

(Fig. 58) has recently been invented into which the *masse cuite* regularly flows without stopping, and which only discharges turbined sugar. The

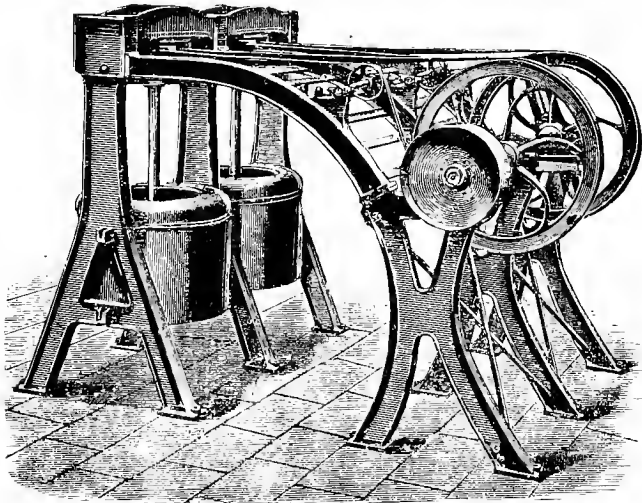


FIG. 57.—Suspended turbines (CAMBRAY.)

principle of this machine is, that the drum, instead of being a cylinder, is a reversed cone. It is fed from the bottom of the cone, and the

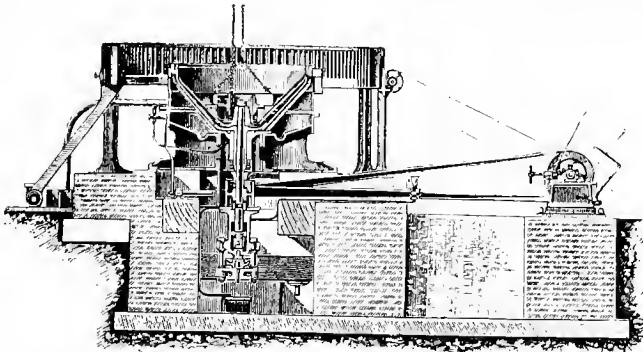


FIG. 58.—Continuous turbine, Szentowsky and Piatkowsky's system (CAIL).

centrifugal force causes the sugar to ascend up the gradually widening conical sides. This invention is due to MM. Szentowsky and

Piatkowsky. At first it was only possible to produce yellow sugar by means of this machine. Whiter sugars are now being obtained. This invention is perhaps rendered even more interesting still by the fact that there is a great future in store for continuous machines, and there is, especially in the case of the turbine, a great advantage to be gained by automatic working which has not hitherto been accomplished.

Plan of the turbine room.—The turbinng of the boiled mass requires a well-planned installation, so as to be done economically; failing which the manual labour is considerable, because the least defect in the arrangements stops all the work, which has then to be done by hand.

The old routine.—Formerly the work of the turbine house was carried on in the following manner. The *masse cuite* collected in the flat tank already described was lifted by pick and shovel—for it is very hard—by men standing in the tank; consequently this was very hard work. At the side of the tank was a mixer, a kind of toothed mill surmounted by a hopper, into which the men threw the boiled mass by shovelfuls. The mixer rendered the mass homogeneous by the aid of a jet of molasses which continuously trickled into the hopper to moisten the mass. Under the mixer was a door, through which its contents were discharged into a basin placed underneath, which held enough to charge one turbine. Two men seized this basin and emptied it into the turbine, and then replaced it under the mixer and filled it again to charge another turbine, and so on.

The recent and more or less automatic methods.—The whole, or the greater part, of this primitive method of working has been replaced by something more automatic. If the flat tank be retained, it is still necessary to have men to shovel the mass. But these, instead of shovelling it directly into the mixer, throw it into a trough placed within their reach, in the bottom of which a helix revolves. The molasses for moistening falls continuously and uniformly into this trough.

The helix conveys the mass into the mixer, and from the mixer it falls into another helix or inclined Archimedean screw, which raises it into a trough placed horizontally behind the long line of turbines and extending all its length. In this trough revolves an axis, furnished with blades, which keeps the mixture uniform.

In front of each turbine a door is inserted in the trough, opening into a gutter, which conveys the moistened mass into the basket of the turbine. When the turbine is charged, the door is closed. But by working thus the charging is done irregularly.

A small measuring tank was then placed between the trough and each turbine, which was filled by opening the door, and closing it when it was perfectly full. It was emptied into the turbine through

another door. A still better arrangement was effected when the two doors were made to work by a single pull with one arm of a lever, the one door only opening when the other door was closed.

The helical systems spoil the grain of the sugar.—All these helical systems work very well, but they are blamed for spoiling the grain.

Remedy.—Therefore, when the plan of the place permits of it, it is preferred to elevate the cooling tank, so that the mixer discharges directly into the distributing tank with its small measuring tanks. This method works well.

Charging by overhead trucks.—In those factories where an under-driven form of turbine is used, entirely open above, small trucks are generally employed, opening underneath and containing the correct charge of one turbine. These trucks run on rails suspended above the line of turbines; they are filled directly under the mixer, are then brought above the turbines, and their contents are all at once emptied into the turbine. This system of trucks is indeed the only one to be recommended when there is a long row of turbines; the helices do not work regularly in such a case. Trucks with side openings have also been built for use with French turbines, and they answer well, although they would not be so convenient with the under-driven turbines. When it is customary to run the boiled mass into the small Schutzenback tanks, the place for the mixer can be chosen; it can be placed on a higher floor to the turbines, and trucks may be used without difficulty.

Discharging the Schutzenback tanks by compressed air.—In emptying these tanks into the mixer, care is taken to leave a small hole in the bottom of the tank, closed by a screw stopper. The tank is brought full above the mixer, and, being held by its wheeled support, it is turned upside down (*i.e.* tilted), the cork is unscrewed, and through this opening a bellows is introduced, which presses behind the mass and forces the whole block right out of the tank. This conical block is seized by the teeth of the mixer and thoroughly incorporated with a little molasses. The work is done well and very rapidly. The bellows which is used is a small air-compression pump, and the nozzle, which fits into the hole in the bottom of the tank, is connected with the pump by a length of indiarubber tubing.

The Bocquin-Lepchinsky mixer and its numerous advantages.—But the most rational process of all is to use the Bocquin-Lepchinsky mixer, already described on p. 243. The well-mixed boiled mass, cooled to a nicety in the vessel, is collected with the greatest of care in the trucks for charging the turbines through a door fixed in the bottom of the mixer. Here (1) workmen are no longer required to extract the mass in the tank; (2) no more destruction of crystals; (3) easy and correct working; (4) a large number of the turbine staff of hands are dispensed with; (5) great cleanliness in the turbine

house,—in fact, everything which one can desire of the best for this very delicate operation. Of all the equipments, this presents the greatest number of advantages; only, the vacuum pans must be elevated one storey further up, which, however, does not cause much trouble.

General method of working turbines.—When the turbine is charged it is generally first started by hand, then the driving strap is put on near the coucs, according to the model of machine, and it is allowed to turn during the number of minutes which correspond with the *degree of purity* which it is desired to obtain in the crystals.

Different grades produced by different treatment in the turbine.—In fact, according to the custom of the market and the commercial skill of the manufacturer, it may be necessary to make extra-white sugar, as in Russia or in France—cube sugar, white sugar, yellow sugar, or brown sugar, all of which qualities require a special turn of the wrist. In making extra-white sugar it is necessary to keep the turbine going for ten minutes; it must be cleansed or purged with water or steam, so as to obtain a hard and compact mass in the turbine.

Cleansing the crystals in the turbine to obtain a very white dry sugar.—Cleansing is an operation which consists in pouring on the mass in motion a certain quantity of pure sugar liquor or water. This liquid displaces the molasses which the centrifugal force cannot detach from the grain. If the cleansing liquor be a sugar syrup, it *takes the place of the molasses*; if it be water it *dissolves a certain quantity of sugar* and acts like the syrup cleanser, which is nothing more nor less than sugar dissolved in water. Cleansing by steam acts in the first place like water, because a portion of the water condenses between the crystals; but when the sugar has reached a certain temperature in consequence of this condensation, the steam, no longer condensing, dries the crystals and frees them from the very fluid, hot, cleansing liquor covering them. There is then found in the turbine a very white and very dry sugar.

Method of cleansing to get an ordinary white sugar.—If an ordinary white sugar be required, a solution of sugar is used less pure than that employed in the first instance. For this purpose that which flows from the turbine at the time of steaming, as a yellow liquid, is used, infinitely purer than the primitive molasses. In that case, cleansing is done by steam. Four to five minutes suffice to obtain this product.

Ordinary yellow sugar.—To make ordinary yellow sugar, they turn three or four minutes. The duration and the nature of the cleansing liquor depend on the percentage test aimed at with the sugar in question. If no cleansing liquor be added at all, brown sugars, giving a low percentage test and still containing a certain amount of molasses, are produced.

Cleansing by steam injector.—It has been attempted to substitute for steam, in the cleansing of the crystals, which dissolves much sugar, a mixture of air and steam by means of a steam injector, bringing a certain amount of air in its train. This system answers well, and is more economical than steam alone. For the number of injectors in use in France for this purpose, see Table LI. p. 250. Hot moist air has also been tried. But these systems are not very general; they do not appear to have any very great advantage over simple steam.

Transferring the turbinéd sugar to the sugar granary.—When it is thought that the *masse cuite* has been sufficiently turbinéd the machine is stopped, and the sugar collected, either by filling it into sacks or by letting it drop under the turbine on to mechanical conveyors; it is then raised on to the sugar granary—an operation which is generally performed by elevator buckets similar to those employed in flour mills, or, when the sugar is in bags, by means of a crane. In the sugar granary the sugar is first dried, then mixed, screened, bagged, and despatched.

Drying the turbinéd sugar.—Extra quality sugars are generally dried by spreading the sugar in thin layers on the floor, and systematically raking it about from time to time. Sometimes the sugar is run into a large revolving cylinder, through which a current of air is passed. When this is done the hot sugar from the turbine is run into it, and it comes out cold and dry.

Method of mixing the sugars.—The mixing of sugar is done by laying it down in heaps, which are turned over by the shovel. In large factories this operation is done mechanically by forming the heap on a trap-door on an elevated floor, which is opened so as to precipitate the sugar on to the floor underneath, from which in its turn it is precipitated on to the next, so that all these successive falls sufficiently mix it.

Screening and bagging up.—The sugar is then screened, to keep out any core, then bagged, and in certain countries lead-sealed by the Excise, after which it is despatched.

“Firsts” sugars and “firsts” syrups.—The molasses which soiled the crystals of sugar in the *masse cuite*, pass, as already mentioned, through the wire gauze of the turbine, are projected against the side of the cover, and issue through a hole made at the bottom of this cover, constituting what is called the syrup from the “firsts” *masse cuite*; the sugar obtained being called “firsts” sugar.

The quality of syrup improves from beginning to end of turbinéd process.—The quality of the syrup varies with the progress of the turbinéd. It is very impure at the outset, since it consists of molasses, but it becomes very fine towards the end. Formerly no heed was taken of this difference, and all the drainings flowed into the same tank—a most wasteful method.

But, later on, these drainings were separated. The first only went to the common tank. The second, which has the same purity as sugar factory syrup, and purer still towards the end of the process, was sent back to the vacuum pan to be boiled, like ordinary syrup. The two are separated by providing the exit with a movable gutter, which the workman directs either into the impure drainage trough or into a parallel one for the pure drainings. This change may even be done automatically at the same time that the steam valve is opened for clarifying, so that the workman does not forget it; for everything which helps the workman and does not leave the work at his mercy avoids errors and mistakes.

During the season 1899–1900, 35 factories in France did not return the drainage syrups or “égouts” for further treatment, 64 returned them to the raw juice or to the carbonatation, whilst 267 returned them to the vacuum pan.

To ensure good and economical working, these drainage liquors are analysed, and those which are pure enough are transferred to the “firsts” vacuum pan, whilst the remainder are sent to the “seconds” pan. The drainage liquors from the “seconds” are almost always rich enough to be reboiled. This is the case when the yield of “firsts” and “seconds” is not very high. With good yields from the “firsts” and “seconds” turbines, the drainage from the “seconds” are quite poor enough to be sold as molasses.

Moreover, the *saline coefficient* of molasses, *i.e.* its sugar divided by its ash, being about 5,—when this figure is reached there is little chance of getting crystallisable “thirds.”

The boiling of low or poor quality products.—*Concentrating and crystallising the syrup from the “firsts.”*—The syrup or drainage from the “firsts” as it flows from the turbine is still capable, under favourable conditions, of producing crystals. This molasses, which moistens the grains in the *masse cuite*, is still very rich, and it becomes richer still by the melting of the sugar during cleansing. It is necessary to filter it to remove any solid and mucilaginous impurities which it may contain, and then to evaporate it to bring it to the strike-point, and then to abandon it in tanks at a temperature of 30° to 40° C. (86° to 104° F.), to enable it to crystallise at the end of a few days.

Method of boiling.—The boiling of the “seconds” is generally done in a special vacuum pan used for this purpose. Sometimes the “seconds” are boiled in the same pan as the “firsts,” between two boilings when there is time. But it is better to use a special pan for this work. The “seconds” *masse cuite* is run into tanks of the same capacity as the vacuum pan. They are arranged in a row, and are sufficient in number to contain the work of two months, the maximum time required for their crystallisation (see Fig. 61 and context, p. 278).

LII.—TABLE OF THE QUANTITIES OF STEAM CONSUMED PER TON OF BEETROOT IN THE DIFFERENT OPERATIONS OF A SUGAR REFINERY WORKING 300 TONS OF BEETROOT IN 24 HOURS (CAMBIER).

Description of Operation.	Kilogrammes of Steam expended in Heating and Evaporating.	Kilogrammes of Steam expended by the Engines and restored in part on Evaporation.	Kilogrammes of Steam restored on Evaporation.	Excess of Consumption of the Motors over Evaporation.	Kilogrammes of Steam really expended without Restitution.	Total Steam expended on Heating, Evaporation, and Motive Power.
Diffusion	107·69	107·69
Carbonatation and saturation	223·73	223·73
Evaporation	426·77	426·77
Heating the syrups and boiling of "firsts"	85·24	85·24
Boiling of "seconds"	25·68	25·68
Motive power	584·211	426·77	25·44	129·84	129·84
Expended in motors without restitution	25·44
	869·11					1024·39

Variations in quality and consistency.—The crystalline mass is sometimes very hard if the “seconds” are rich, sometimes mushy if they are poor. That depends on the yield of the “firsts,” because if the “firsts” are abundant, as they should be, there will be but little “seconds.” These differences originate in general in the good or bad management of the factory, and are also due sometimes to the quality of the beets which have been wrought.

The “seconds” *masse cuite* crystallised in the tanks is turbinéd, and gives “seconds” sugars, which are yellow. The drainage from the turbines ought to be reboiled, again run into the crystallising tank, and afterwards turbinéd. It yields “thirds.” It is rarely that “fourths” are made in *beet* sugar manufacture.

The time required for the crystallisation of the “thirds” is much longer than for the “seconds.” It requires at least six months. They are therefore very often not turbinéd until the factory starts work again in the following season, so as not to resume this work in the summer, bringing workers back expressly and the whole of the staff of the factory.

The fill-houses.—The “firsts” and “seconds” tanks are contained in vast buildings called fill-houses. These fill-houses occupy the whole of the ground floor of these buildings, and the upper floor is generally occupied by the sugar warehouses, which should be correspondingly large.

Temperature of the fill-houses.—The fill-houses ought to be continually kept at a temperature of 40° C. (104° F.). This temperature is obtained either by steam heating or calorifers, or by kinds of *braseiro* (braziers), which burn night and day in the rooms; calorifers heated from the outside being the most practical. However, during manufacturing, steam heat is generally used, and there is also great economy in utilising the steam from No. 1 or No. 2 vessel of the triple-effect plant to get the temperature required for the heating of the “seconds.”

MANUFACTURE OF BROWN SUGAR

The more pure the *masse cuite* is, and the more impure the syrup separated from it centrifugally, the greater is its yield in sugar.

In the old system of working (large or small boiling vessels) it is generally impossible to lower the purity of the discharge below 75 per cent. of real sugar. But since *crystallisation in motion* (see pp. 243, 255, and 279) has come into favour, this purity may, within certain limits, be lowered at will. All that has to be done is to sufficiently concentrate the *masse cuite*, and then to keep it in motion a certain time at a given temperature. There is thus obtained much greater yields of “firsts.”

But up to what point is this increase remunerative? To elucidate this question Dr. Classen has prepared two tables.

Table LIII. shows the influence of the increasing purity of the boiled mass on the yield when the discharge is of constant purity; whilst Table LIV. shows the variations in yield with constant purity of the boiled mass, but with variable purity of the discharge. Table LIII. has been calculated on the supposition of 6 per cent. of moisture in the *masse cuite*, say 94 per cent. of dry matter. The sugar extracted is supposed to be 96 per cent. polarisation, 2 per cent. of water, 2 per cent. of non-saccharine, 98 per cent. of purity; the discharge has a purity of 72. The amount of yield is determined according to Hulla's formula—

$$\text{Sugar per cent.} = \frac{S^m C^p - E^p}{S^m (S^p - E^p)}$$

In this formula C^m = dry matter per cent. of boiled mass; S^m = dry matter per cent. of turbinated sugar; C^p , S^p , E^p = the quotients of purity respectively of the *masse cuite*, turbinated sugar, and the green discharge (quotient of purity = ratio of sugar to total dry matter).

TABLE LIII.—INFLUENCE OF PURITY OF THE “MASSE CUITE” ON THE YIELD IN SUGAR WHEN THE DISCHARGE SYRUP HAS A CONSTANT PURITY OF 72.

“Masse Cuite.”		Yield of Crude Sugar per cent. of “Masse Cuite.”	Increase of Yield per each cent. of Purity.
Dry Matter.	Purity.		
94	88	59.0	3.7
94	89	62.7	3.7
94	90	66.4	3.7
94	91	70.1	3.7
94	92	73.8	3.7
94	93	77.5	3.7
94	94	81.2	3.7
94	95	84.9	3.7

In Table LIV. the *masse cuite* has been taken as containing 94 per cent. of dry matter and 91 per cent. of purity, sugar at 98 per cent. dry matter and 98 per cent. of purity; the discharged syrup decreasing in purity from 75 to 66.

TABLE LIV.—INFLUENCE OF THE PURITY OF THE DISCHARGE ON THE YIELD IN SUGAR OF THE BOILED MASS WHEN THE PURITY OF THE LATTER IS CONSTANT.

Purity of Discharged Syrup.	Yield per cent. of the Boiled Mass in Raw Sugar.	Increase of Yield by a Diminution in Purity of 1 per cent.
75	66·7	...
74	67·9	1·2
73	69·1	1·2
72	70·1	1·0
71	71·0	0·9
70	71·9	0·9
69	72·7	0·8
68	73·5	0·8
67	74·2	0·7
66	74·9	0·7

According to Table LIII., for an increase of 1 per cent. on the purity of the boiled mass, other conditions being granted, the yield of the *masse cuite* increases 3·7 per cent. According to Table LIV., a lowering of the purity in the discharged syrup of 1 per cent. only leads to an increased yield of raw sugar of 1·2 to 0·7 per cent. of raw sugar in the boiled mass. This is easily explained, since the weight of the discharged syrup hardly represents one-third of the boiled mass.

The increase in purity of the *masse cuite* should therefore be the best method of obtaining greater yields. Unfortunately this method is not practicable, supposing always that we employ the same methods as in the past—diffusion, defecation, and saturation. The purity of the *masse cuite* depends therefore more especially on the beetroot, and consequently it is a factor which varies from year to year.

On the other hand, discharge syrups of a low degree of purity may always be obtained by resorting to *crystallisation in motion*.

It remains to be seen up to what point it is advantageous to lower this coefficient. Two principal factors are to be considered in this operation—the duration of the agitation, and the quality of the product obtained.

Experience has demonstrated that it is possible in a short time, generally from 12 to 24 hours, to reduce the purity of the discharge from 75 to about 70, but that each unit gained below this limit requires a greater and greater loss of time, and consequently involves a correspondingly increased extension of plant.

As far as the quality of the sugar is concerned, the product of

crystallisation in motion is better than that of turbinizing, after allowing the boiled mass to stand in the crystallising tanks. The sugar of *crystallisation in motion* consists of larger and more regular crystals, and it contains a more fluid syrup. If the agitation be prolonged so as to lower the quotient of purity of the discharged syrup below 70, more and more sugar will crystallise, which in ordinary working would have been obtained in the "seconds." Now this sugar is not so white as that which crystallises during *boiling in vacuo*, and at the beginning of the agitation of the *masse cuite* in the crystallising vessel, and it is so much greyer or browner in tint the lower the purity of the surrounding syrup has been reduced.

Thus, whilst the nucleus of the crystals consists of sugar absolutely white, the outside layers are coloured to a greater and greater extent. Naturally, the coloration depends greatly on the shade of the concentrated juice from which the boiled mass was produced. Bad dark juices, especially those resulting from unripe or diseased beetroots, often yield crystals absolutely deprived of whiteness. But, even in the case of very white syrups, the sugar which crystallises from discharges of less than a purity of 70 is not white. Now, as the first condition for a good "firsts" sugar is to produce, by refining, crystals as white as possible, it is evident that in the manufacture of these sugars it is necessary to avoid prolonging the crystallisation in motion for too great a time.

A purity of 70 to 72 ought therefore to be regarded as normal in the working of "firsts" *masses cuites* by crystallisation in motion, so much the more as each unit of purity corresponds to 0.7 to 0.8 per cent. of increased yield—a gain which would not appear to be in proportion to the loss of time and diminution of quality in the product. If it be desired to apply crystallisation in motion to inferior quality syrups, it would be preferable to work up these syrups by themselves with remelted low quality sugars.

Classen (p. 261) states that Hulla's formula is perfectly applicable to crystallisation in motion. The syrup added to the *masse cuite* must also be taken into consideration as an indifferent body, having, however, two conditions to fulfil. The first condition is to mix with the primitive syrup, so as to prevent the latter becoming too concentrated, and thus give birth to fine crystals instead of enlarging the crystals already formed. The second is that the syrup added fixes any water which may be in excess for the crystallisation of the syrup of the boiled mass, and would finally be opposed to the complete crystallisation of the latter, except for the purity of the added syrup. In fact, the added syrup has no influence on the increase of yield of the *masse cuite*, and may therefore be left out of account.

It is true that in working, by adding syrup towards the end of the boiling, according to Bock's process, the composition of the *masse cuite* is unknown. In that case the purity of the mass may be

determined from that of the concentrated juice of the beetroot as identical with the latter, if the slight loss of sugar due to heating be deducted. The quantity of water in the boiled mass still remains to be ascertained. But it may be taken in all cases as 5 or 6 per cent., according to what it formerly was. With these elements of the composition of the sugar, as well as the purity of the syrup, being easily determined directly, Hulla's formula may be applied.

Example.—Average of one week: purity of syrup added to the boiling, 92; composition of the sugar—96 per cent. polarisation, 2 per cent. water, and 98 purity; purity of the turbined syrup, 72; water in the *masse cuite*, 5 per cent.

The yield of raw sugar of the *masse cuite* in weight—

$$\frac{95(92 - 72)}{98(98 - 72)}100 = 74.5 \text{ per cent.}$$

SUGAR WAREHOUSES

All the "firsts" sugars, as already mentioned (see p. 257), are mixed in the warehouses. The sampling and bagging-up are done there. The bags are all weighed and lead-sealed by the Excise in certain countries. Generally this class of sugar is not stored in the warehouse; if it be not sold it is sent to the *depôt*, where it is received, against a negotiable receipt, which facilitates trade without there being any necessity to touch the stored sugar.

The "seconds" are sent to market in the same way; but the "thirds" remain a longer time in the store, owing to their tardy manufacture. In those countries where only yellow sugar is made, even from the "firsts," sometimes the "firsts" and the "seconds" are mixed according to the percentage of sugar wanted.

In Russia, where only extra-white sugars are sold, the "seconds" and the "thirds" remain in the warehouse, to be remelted next year, so as to get nothing but white sugar.

Capacity and function of warehouses.—The sugar warehouse is therefore the place where the commercial part of the factory is carried on, and should be capacious enough to contain a stock of manufactured products corresponding to the work of the factory, if the conditions of sale or storage in the *depôt* do not appear satisfactory to the manufacturer, if the market price is too low, or the charges too high. These vary with the country, the harvests, and the temperament of the directors. There is no general rule upon the point.

MOLASSES

Beet-sugar molasses.—*Their nature.*—The drainings from the turbinng of the last, *i.e.* the lowest-quality sugars, constitute the molasses properly so called. These molasses still contain 50 per

cent. of sugar. But the remainder of the substances in solution consist of organic and saline impurities, which prevent crystallisation of the sugar, and which are consequently termed *molasses-producing bodies* (*matières mélassigènes*).

Disposal and packing.—The molasses are collected in large reservoirs during the season, in vats, tanks, or cisterns, then run into casks (petroleum barrels are generally used), and sold to the distilleries.

Their sugar content.—As in general 3 per cent. of molasses are obtained in beet-sugar factories, there is therefore $1\frac{1}{2}$ per cent. of the original sugar still remaining in them (see detailed composition and method of analysing molasses elsewhere).

Attempts to recover sugar.—Accordingly different methods have been tried to extract the sugar from the molasses, and this is sometimes done economically. Unfortunately the barbarous legislation which regulates the tax on sugar in some countries stifles any movement in this direction made by sugar manufacturers, under the pretext that they would thus produce sugar free of duty. The different processes proposed, adopted, then abandoned under the unjust taxes of fiscal systems, then resumed under more equitable legislation, have not become general, so that the factories which extract sugar from molasses, or have an improved system in the working of which no molasses are made at all, are very rare.

Abolition of molasses liberates vast working space now occupied by fill-houses.—However, the abolition of molasses is a very important point in sugar factories, because it increases the yield, and does away at the same time not only with the long, tedious, and clumsy work of the fill-houses, but with the fill-houses themselves, freeing in this way the vast spaces used for the crystallisation of low-quality products.

The different processes proposed for this are legion. Those which have survived the test of actual working, and which are met with in those privileged factories which have been able, either by means of abundance of available capital or through the more enlightened intelligence of their owner, to transform their method of working so as to abolish molasses, specially claim attention.

The old alcoholic method.—The old process suggested was the use of alcohol. If alcohol be mixed with molasses the major portion of the organic products dissolve, whilst the sugar, much more insoluble, is precipitated.

The lime process.—Another very old process consists in mixing the molasses with lime. A solid magma is formed, a kind of hard mortar formed by the combination of the lime with the sugar, under the form of an *insoluble tribasic sucrate*. By washing this magma with water or alcohol an insoluble sucrate of lime is obtained, and a liquid containing the greater portion of the impurities in the state of lime salts.

In both these systems much sugar was lost in the wash-liquors. Those proposed since are only improvements—the practical perfecting of those old experiments with which the names of several celebrated chemists are associated, such as *Pesiér*, *Marguerité*, and others.

The osmogène.—Finally, Dubrunfaut proposed and used a system based on the osmotic action of membranes (see pp. 79–81), to separate the sugar in the molasses from the saline molasses-producing substances, which prevent crystallisation. The osmose process became famous, and reigned gloriously for a certain time; but the fiscal system killed it, and it has not been born again, in spite of the efforts of Leplay and the remarkable improvements he has brought to bear upon it. However, some factories still have their osmogènes, and still use them when the harvest, the price of sugar, or the fiscal difficulties permit, in such a way that they must be regarded, as Horsin-Deon humorously points out, as amongst the living animals, like the dormouse, which wake up from time to time as fine weather comes round (see Table LV. p. 268).

The theory of osmosis or dialysis.—When describing the diffusion process, the theory of the phenomenon termed osmose was explained—a phenomenon which induces the passage of two currents in a contrary direction through a membrane when its faces are in contact with solutions of different densities.

This phenomenon, which occurs in plants through organised tissues, was tested by Graham on non-vegetable membranes or septa of animal origin, e.g. bladders, in the apparatus termed endosmometers.

Parchment paper as a membrane.—Dubrunfaut applied the osmotic property to molasses by using parchment paper as a membrane. He noted the following results:—

If on one side of the membrane there be placed pure water and on the other side molasses, *a current of water passes to the molasses, whilst a saline current passes from the molasses to the water.* This saline current contains much salts, some organic matter, and a little sugar. In the beginning salts pass almost alone, later on the liquid contains a little sugar, still later the sugar passes in greater abundance.

If, therefore, the time of contact between the water on the one hand and the molasses on the other be limited, it is possible to extract from that molasses a large proportion of its salts and almost no sugar. Consequently the osmosed mass becomes crystallisable, and may thus be made to give up the sugar which the salts with which it was impregnated hitherto kept in solution.

Description of Dubrunfaut's osmogène.—It was to accomplish this that Dubrunfaut constructed his *osmogène*. It consists essentially of a series of parallel chambers in juxtaposition, having the form of a thin parallelogram, in which water circulates in the one and molasses in the other, the whole being kept hot, for *osmosis is much more rapid when the liquids are hot than when they are cold.*

The molasses chambers are intercalated between the water chambers, one between every two. The liquid enters below and flows upwards, following a zigzag movement induced by divisions made in the frames. In the parallel chamber the liquid follows the reverse direction. All the frames work separately, and thus form elementary osmogènes joined side by side into a single machine. By suitably regulating the molasses and the water-supply taps, the osmosis may be pushed to any desired point.

As the water becomes charged with salts it increases in density.—On the other hand, *the molasses diluted with water diminishes in density.* The apparatus is regulated by taking the density of the two liquids at their exits, after having found by analysis the density at which to stop. Osmosed molasses and exosmosed water are thus obtained. In order to purify the molasses as much as possible, the osmosis must be pushed rather far. Therefore the exosmosed water contains much sugar. If it be concentrated and re-osmosed, re-osmosed molasses are obtained—which may again be crystallised—together with fresh exosmosed water, which may again be osmosed repeatedly. Three or four successive osmoses are made in this way, and a very saline molasses is finally obtained which still contains sugar, but which is used more especially in the manufacture of potash salts. Most of the potash used in France in soft-soap manufacture is obtained from molasses.

The osmogène made of wood is cheap, and is of great service when it is known how to take advantage of all its capabilities. Unfortunately, legislation has banished it from France. Some are still in existence in Belgium and Russia.

Leplay's improvements on the osmogène.—Leplay, the worthy successor of Dubrunfaut, has made some improvements on the osmogène. The great defect in the osmogène is the change of density of the molasses, which enters at a high density and issues diluted with water, so that in the frames the density of the liquid is greater below than above. But as osmose only goes on at a certain density, the purification is likewise better at the bottom than at the top. One portion of the surface of the frames is therefore, so to speak, lost.

Evaporative osmogènes.—Leplay has therefore constructed osmogène-evaporations, which consist of ordinary osmogènes surmounted by a basin with a steam coil, in which the osmosed molasses continuously returns, and is brought back to its original density. This basin is formed by the frames themselves, which, pressed against each other, form on the top a plane surface with edges formed by the prolongation of the mountings of the frames.

The evaporating osmogène has a considerable osmotic capacity, and yields remarkable products, both as regards richness and uniformity.

LV.—TABLE SHOWING THE GRADUAL DECLINE TO PRACTICAL EXTINCTION OF USE OF OSMOGENES IN FRANCE.

Season.	Number of Factories possessing Osmogènes.	Number of Osmogènes.	Total number of Frames composing the Osmogènes.
1884-85 . .	154	1085	74,783
1885-86 . .	221	1458	97,789
1886-87 . .	289	1884	149,086
1887-88 . .	223	1502	124,839
1888-89 . .	39	316	29,867
1889-90 . .	5	34	2,935
1890-91 . .	1	4	400
1891-92 . .	2	44	4,440
1892-93 . .	1	4	400
1893-94 . .	2	56	2,925
1894-95 . .	1	50	2,550
1895-96 . .	1	50	2,550
1896-97 . .	6	...	3,830
1897-98 . .	4	...	4,140
1898-99 . .	1	...	2,550
1899-1900

Steffen's sucrate of lime separation process.—The only process which has, under the name of the separation process, survived the fiscal red-tapeism is that of Steffen, an Austrian. It is used in only a few factories, although it succeeds admirably with those who know how to work it. Unfortunately its initial installation runs rather dear. But manufacturers who have enough capital to pay for this lucrative luxury find it to answer well.

It consists in treating the molasses in the cold by powdered quicklime. When the degree of dilution is well observed and the temperature sufficiently low, below 15° C. (59°·2 F.), and the lime good, an *insoluble sucrate of lime* is formed which contains a little more lime than the *tribasic sucrate*, and the impurities remain in the liquid. A liquid mud is thus obtained, which is filtered, pressed, and washed in the press, thus bearing sucrate of lime therein, and a mother-liquor which contains all the salts, organic bodies, and a little dissolved sucrate of lime.

Separation of the sugar from the sucrate of lime by carbonatation.—There are two methods of separating the sugar from the sucrate of lime. One consists in mixing the insoluble sucrate with water and carbonatating it. This is not a good system, owing to the difficulty of effecting carbonatation of the insoluble sucrate under such circumstances.

The solution process.—The other method consists in dissolving

the sucrate in a sugar liquor called the solution process. *Soluble monobasic sucrate* of lime is formed which dissolves in the liquid, and the excess of lime is precipitated. They are separated by the filter press, and the liquid carbonated. But the solution system is not wrought thus by itself, but with the ordinary carbonation of the juice.

Dovetailing the recovering of sugar from molasses into the carbonation process.—Generally the molasses are wrought up during the period of treating the beets. Saccharate of lime is produced in an annexe of the factory from the molasses, and the saccharate is employed in both the first and second carbonations in place of lime. Both carbonation and solution are effected simultaneously, the juice is enriched in sugar, and no special plant is required for treating the saccharate. If only a quantity of molasses, corresponding to the lime required for the liming of the juice, be treated by the separation process, very good results are obtained.

A portion only of the molasses can be advantageously used up in carbonation.—But sometimes the recovery of the sugar from the molasses is pushed so as to extract more sugar. Then the saccharate, which still contains organic impurities, whatever care be taken in washing, modifies the composition of the juice, and the factory works badly. A check is thus courted by the production of impure syrups.

Supplementary method.—It is far better, if there be an excess of saccharate left over, to dissolve it with the juice of the second carbonation, and to pass it to the filter presses to separate the excess of lime, and then to carbonate those liquids. It gives more trouble, but it is safer. The best process is not to convert more molasses into saccharate than the safe treatment of the juice necessitates. (For chemistry of sucates, see p. 122 *et seq.*)

The details of Steffen's separation plant.—The essential part of the separation plant consists of a *refrigerating mixer* (Fig. 59). This is a tubular body which exactly resembles a vertical triple-effect vessel, jacketed; cold water—as cold as possible—circulates in the jacket and in the intertubular space. It is surmounted by a hopper, from which it is separated by a revolving measurer, for introducing the powdered quicklime. A strong agitator keeps the liquid in motion.

Grinding the lime underneath flat stones.—A crusher near the lime kilns reduces the lime to coarse grains. This granulated lime is then ground underneath flat stones like those of a flour mill, then passed into sifting machines, from which it issues in the state of a very fine impalpable dust. In this part of the factory one would think they were in a mill, if only for the lime dust which flies about somewhat in every direction. It is this lime flour which is introduced into the refrigerating mixer.

Weighing the molasses.—On the other hand, into a tank placed

on a weighing machine the amount of molasses to be introduced into the mixer is carefully weighed, whilst the water required for dilution

flows in until it reaches the desired level. There is thus about 3000 litres of mixture.

Measuring the powdered lime and feeding into the machine.—

Mixing.—When the mixer is full and at work the lime is added by means of the measurer. Each time that a measure of lime is introduced the temperature rises, and it is allowed to fall to 12° to 13° C. before the next addition of lime. When the density of the liquid shows that no more lime is required, the machine is allowed to make a few more turns and the operation is finished.

Filter-pressing the mixed produce and converting it into sucrate milk.—The contents of the mixer are then passed to the filter presses, washed, the cakes placed in another mixer, where they are mixed with water, and the resulting sucrate milk is used for carbonatation instead of milk of lime.

Treating the mother-liquors.—The mother-liquors from the filter presses still contain a little sugar. They are brought to the boil. *Tribasic sucrate* is formed, which is collected in the filter presses and added to the sucrate.

The above processes collectively form the separation process, the very simple working of which renders it very practicable. It, however, requires sustained attention and a continuous analysis of molasses. The works chemist is the mainstay of this operation,

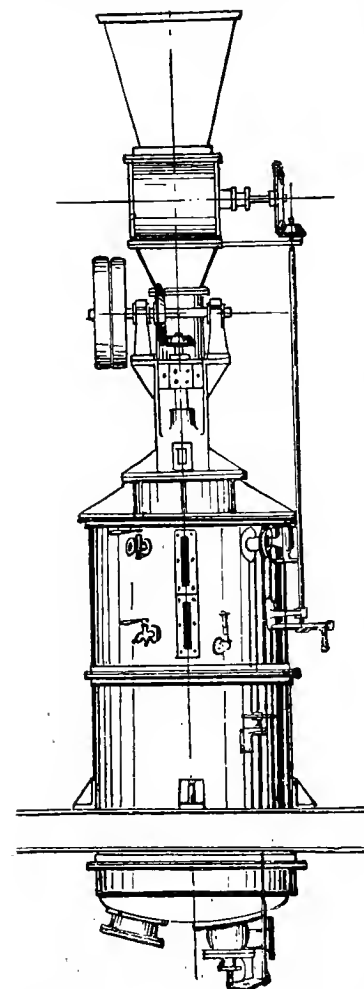


FIG. 59.—Refrigerating mixer used in the separation process (F. OLIN).

the process varying with the raw material used.

The alcoholic method of extracting sugar from molasses.—Man-

oury's process.—The alcoholic processes of extracting sugar from molasses, already referred to on p. 265, have nearly all been abandoned. There, however, remain a few applications of Manoury's system in Germany and Russia which require special mention.

Treats the molasses with lime and elutriates with alcohol.—Manoury's process consists in preparing a solid combination of the molasses with lime in a granular form, and in treating the granular mass with alcohol in diffusers or elutriators.

Description of process.—The special form of calcic compound formed with the molasses is obtained by adding to the molasses, contained in a horizontal mixer, powdered slaked lime obtained by dipping lime in lumps into water, and allowing it to slake without any further addition of water. The molasso-calcic granules thus obtained are placed in the elutriators. There are four of them, each containing 8 tons. Then alcohol is circulated so that the special diffusion lasts 24 hours. For 100 kilogrammes of molasses 294 litres of 40 per cent. alcohol (say 29·4 gallons per 100 lbs.) are used.

Rectification of the alcohol to obtain residual sucrate.—The alcohol, having done its work, is rectified. That remaining in the elutriators is driven over by steam, and likewise collected. There then remains in the vessels a *sucrate mud* fit for use in carbonatation.

Loss of alcohol in process.—The loss of alcohol is the chief difficulty. However, Manoury has so well organised his plant that the leakage of 40 per cent. alcohol is only 2 per cent. Well-equipped factories which have erected this system have preserved it in spite of new methods invented too soon to have allowed the alcohol process to become to any extent general.

The strontia method of extracting sugar from molasses.—Another process which was very much in vogue, and which still exists in Germany, is the extraction of sugar from molasses by strontia. Though the *Raffinerie Parisienne* still works by this process, strontia is too dear for it to become general.

Advantages of sucrate of strontia over sucrate of lime.—Strontia sucrate is much more easily formed than lime sucrate. That is why the celebrated German chemist Scheibler has pressed its use in sugar factories, the more so as Germany has extensive strontia deposits, where it can therefore be obtained very cheaply.

Description of the strontia method.—The operation is very simple. In a vessel, cooled to the surrounding temperature, the molasses are mixed in suitable proportions with the strontia. Sucrate of strontia is formed, the mother-liquors are separated by filter presses, the cakes washed in the press with cold water, and sucrate of strontia cakes are thus formed, which are afterwards mixed with water and carbonatated. The carbonate of strontia is returned to the caustic condition and used over again. All this can be very rapidly described, but the application of this principle is tedious. The mother-

liquors have to be worked up, for they still contain strontia and sugar. The reburning of the strontia is not convenient; the operation as a whole requires extensive plant, because it is necessary to make the strontia and to reburn it to save expense.

Tends to be superseded by Steffen's process.—However that may be, the strontia process has its advocates, although much less is heard

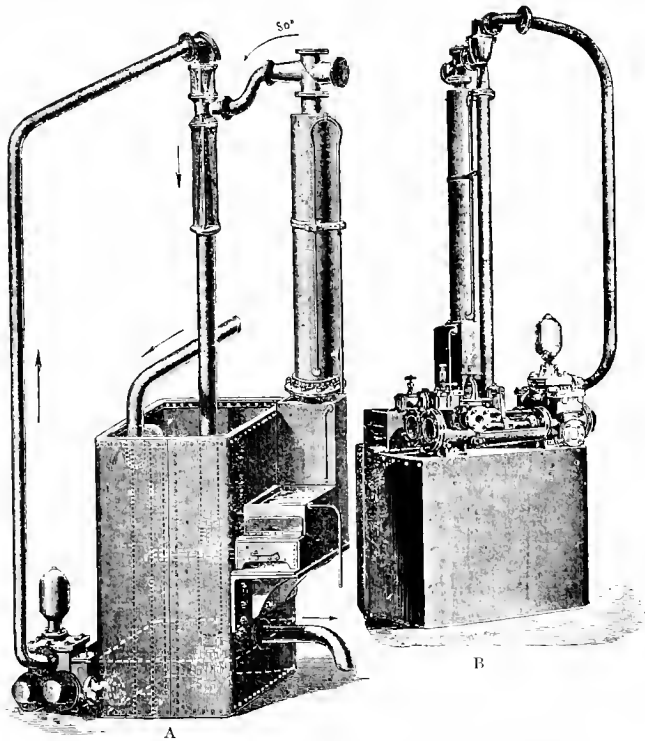


FIG. 60.—Quarez's system of continuous sulphitation (MAGUIN).

of it since the introduction of Steffen's process, which presents many advantages over it.

Influence of continuous sulphitation of the syrups and drainage liquors on the viscosity of molasses.—The practice in sugar factories of returning the drainage liquors to the "firsts" vacuum pans and the different processes leading to the production of molasses in manufacture have demonstrated the necessity of eliminating as far as possible the viscosity of the "firsts" and "seconds" syrups. The

most general method is to treat them with sulphurous acid. The Quarez system (Fig. 60) presents numerous advantages:—

1. The sulphurous acid is drawn into the syrups by aspiration, and not forced into them by compression.

2. The quantity of syrup in contact with the sulphurous acid is very small, 3 to 4 hectolitres only can undergo a false manipulation.

3. The entrance and exit of the syrups being continuous, the sulphitation may be stopped or started instantaneously; the work of the factory can never be stopped thereby, the syrups passing through the apparatus whether sulphited or not.

4. The apparatus may be very easily installed, occupies but little space, and leads itself to every combination of working.

5. Any workman, even a child, can work the machine.

6. It has no valve in contact with sulphurous acid.

Fig. 60, B, shows the apparatus used for the syrups of a beet-sugar factory working up to 300 tons a day, the furnace and the pump on the sulphitation tank itself. The necessary space is reduced to 4 feet by 4 feet.

Fig. 60, A, shows the apparatus employed for drainage liquors, and in the case of factories producing more than 300 tons. The trunk is replaced by a special injector of great capacity, and the tank is higher to allow for the expansion of the froth which is produced so abundantly in the sulphitation of the drainage liquors. The furnace and the pump are then placed either on the floor or at a sufficient height. The apparatus is simply regulated by the speed of the pump, which is increased or decreased according as the sulphite workman finds it more or less alkaline to litmus paper. The quantity of sulphur burnt is in proportion to the draught induced by the passage of the syrups into the injector, and consequently the speed of the pump. According to the output of the factory the apparatus is made with two, three, four, or more injectors, and even in this case the necessary space is reduced to a minimum, and nothing in comparison with the encumbrances incidental to other processes.

During the season 1899–1900 sulphurous acid was used in no less than 162 sugar-beet factories in France in the *purification and defecation of the juice*.

THE ELIMINATION OR AVOIDANCE OF MOLASSES-MAKING IN BEET-SUGAR MANUFACTURE

Crystallisation in motion.—On the principle that prevention is better than cure, rather than try to extract sugar from molasses it seems natural to strive not to so degenerate the sugar as to produce molasses. As molasses is the result of the non-crystallisation of the sugar under the so-called molasses-producing influence of the organic and saline impurities in the juice, the problem therefore is how to

eliminate these impurities. *Carbonatation* eliminates the greatest possible bulk of the organic matter and some saline impurities, but the bulk of the salts remain behind in the juice. Dubrunfaut proposed the previous *osmosis* of the juice to eliminate from it the bulk of the saline matter, but this osmosis, however little, *lost too much sugar*. Up to now, therefore, it has not been found possible to eliminate all the organic matter, far less the salts. If it be not practicable not to make any more molasses, *it is at any rate necessary to find means for making the sugar crystallise in the midst of its enemies, the molasses-producing bodies*. Then molasses will still be produced, but in such trifling quantity as not to be worth mentioning.

Two methods have lately been tried to reach this end. One of these, *crystallisation in motion* (Fig. 62, p. 279), permits this principle to be applied in all factories without at all changing the usual routine—a few more engines, that is all. The other—*Steffen's new process*—changes the whole of the final part of the work, starting from the boiling, and involves extensive and dear plant. But it has the advantage of making all the extractible sugar white, whilst the first method makes as many grades of sugar as it does vacuum pan strikes. When a saccharine solution is left to itself at a temperature of 30° to 40° C., and sufficiently concentrated, say about 40° B. (sp. gr. 1·376), this syrup crystallises slowly. Each crystal formed is fed, at the expense of the surrounding solution, which gets weaker and weaker. After a certain time the solution is too weak, and abandons no more sugar. The sugar and foreign matters are then in the ratio of 3 or 4 to 1. Dubrunfaut said that 1 part of saline matter prevented 3·75 of sugar from crystallising.

Feeding the crystals.—Some years back it was observed that in stirring syrup, in presence of a certain quantity of sugar, in the form of crystals added as a bait or nucleus, and cooling the syrup, the affinity between the sugar and the molasses-producing agents was greatly reduced, and that the yield in crystals was therefore greater. Every one knows, in fact, that when sugar syrup is stirred with a spatula the syrup rapidly crystallises into a compact mass. This was, in fact, the only way the Indians used to crystallise their crudely made and roughly evaporated cane juice. Taking advantage of this fact, a new process of crystallisation in beet-sugar factories has quickly been established. Bocquin and Lepchinsky invented a machine for quickly cooling the “firsts” *masse cuite* before turbinating—a machine which has already been described (pp. 243, 255). It was adopted almost simultaneously both in Germany and Belgium (see also Fig. 62 and context).

Reciprocal crystallising effect of the “masse cuite” syrup and the feeding syrup.—Working upon the fact that syrup crystallises quicker and exhausts itself of sugar to a greater extent when agitated, poorer syrups were poured on to the “firsts” *masse cuite* and stirred

by an agitator; the poorer syrups gave up their sugar to the crystals already formed, in such a way that both the syrup of the boiled mass and the syrup which was thus added were simultaneously exhausted of sugar.

Therefore, if this new mass be turbinated a much greater yield of white "firsts" is obtained than in the usual way of working, and to make additional "firsts" is a step in advance in beet-sugar factories. It is the molasses coming from the turbinating of the "firsts" that is added to the next *masse cuite*. So that, if the process went on indefinitely, the final result would be "firsts" and molasses.

But this syrup very soon becomes too impure. The molasses is then liquefied, either all at once or successively, and boiled as "seconds," which is crystallised in the same way. Finally, a very poor "thirds" is obtained and a very poor molasses. *Crystallisation in motion* is being more generally adopted, but it is still in its infancy.

Steffen's systematic process for reducing molasses production to a minimum.—This process was in use in France in three factories in 1899–1900. It was invented by an Austrian, a very keen observer of very great merit, named Steffen. The process consists of a system of boiling in grain, and treating the boiled mass so as to convert all the sugar into "firsts" sugar, and only to make but the minimum quantity possible of molasses.

The process entails the alteration of the whole treatment of the juice after it comes from the triple effect.—From the boiling of the syrup as it issues from the triple-effect vessel the ordinary method of manufacture is altered.

Feeding the boiled mass with fresh rich syrup.—Steffen observed that the first crystals formed in the vacuum pan are produced in the midst of a liquid, in which the ratio of the sugar to the non-sugar is considerable. But as soon as the crystals are formed the medium in which they swim becomes poorer and poorer in sugar. To feed the *masse cuite* with fresh rich syrup is to change the mode of existence of the crystal, to induce the birth of new crystals, which one is obliged to melt again in reheating the mass; it is, in fact, to bring about a perturbation in that which is already done. Rationally, it would be better to feed the *masse cuite* with poorer and poorer syrups of the same purity as the syrup existing in presence of the sugar at the moment of feeding. This is what Steffen does in his methodical boiling. But in order to arrive at these results it is necessary to have syrups of graduated richness. The method of obtaining these will be given in the sequel. If the mass be fed with poorer and poorer syrups the last syrup will be nothing but molasses. Therefore, if this mass be turbinated, nothing is obtained but white sugar and poor molasses, which there is no need to reboil, because it would yield no more sugar.

By a single operation, therefore, in the vacuum pan it has been found possible to make white sugar and molasses, suppressing the working of the poorer products, fill-houses, etc. The manufacture finished, the factory may be closed. There is nothing further to be done until next season.

Supersedes turbines by methodical washing.—In order to obtain his graded syrups, Steffen does away with the turbines, and replaces them by a methodical washing of the *masse cuite*. This washing consists in cleansing the mass in special vessels with cleansing liquids of a greater and greater degree of purity, which mutually follow each other, until at last only pure sugar syrup is used as the last cleansing agent. Therefore the *masse cuite* cleansed in this way, drained and dried, is nothing less than very pure white sugar.

As these successive cleansing liquids get more and more bulky, the excess is used to feed the vacuum pan.

The molasses removed by exhaust from wire-gauze-jacketed tanks.—The process is conducted in the following way. The *masse cuite* is run into a series of jacketed flat tanks on wheels; the jacket consists of wire gauze analogous to that used in the turbines. First of all, an exhaust forces the molasses which soils the crystals to rapidly pass through the jacket. This molasses is the final product, and is sold to the distillers.

Further cleansing with sugar syrup.—When the molasses has been eliminated a syrup richer in sugar is poured on the mass, then a richer still, and so on to a pure sugar syrup.

The methodical using up of the spent sugar syrup cleansing fluids.—The second cleansing syrup passed is weakened by being mixed with the molasses remaining between the crystals; it may be used as the first cleansing liquor of the following operation. The third cleansing liquor becomes the second, and finally the sugar solution will become the second last.

The storage of the various cleansing liquors.—All these successive cleansing liquors are stored in a large tank, with compartments fitted so that the different liquors flow simultaneously on all the waggons at one operation; all that has now to be done is to empty the waggons, which no longer contain anything but white sugar. This emptying is done by tilting them upside down into a hopper, where there is a mixer to impart a little more homogeneity to the sugar.

Turbining and drying.—From the mixer the sugar falls into the turbines, where a cleansing by dry steam dries the crystals; if this be not sufficient to dry the sugar, it is run into a large horizontal cylinder revolving on its axis, through which a current of air from an air propellor terminates the drying.

Instalment expensive, but gives marvellous results.—Steffen's process works very regularly; it looks, says Horsin-Deon, as if it

were going to be the process of the future. Unfortunately it is very dear to instal. But the results which it gives are none the less marvellous. It has therefore been adopted in some factories, in spite of the sum which has to be disbursed as cost of plant and royalty for use of patent.

Combined with the inventor's separation process, is still more costly and difficult to redeem.—To complete this system and to win from it every possible advantage, it must be supplemented by Steffen's separation process previously described. Because the process now described yields finally as much molasses as the ordinary process with simple turbinng, working in the manner indicated, an extraordinary yield of sugar is obtained, all of which is white, with an insignificant residue of molasses. But by the addition of the separation process the method becomes still more costly, and it is a long time before such a large amount of capital is redeemed. That is the cause of the delay which manufacturers exhibit in adopting these new inventions, whatever profit they might gain by getting all their sugar as white sugar, and by being able to stop work the day the last beetroot is passed, and by abolishing the fill-houses and all the expensive work incidental to the "firsts" and "seconds." Nevertheless, it is a great point gained, and no small consolation, to know that, the day the price of the necessary plant and machinery will permit of it, all beet-sugar factories will be able to work in a much more rational manner than they do now; and, moreover, when the question has been better examined, and thus becomes better understood, means may be found to simplify all these operations and to reduce this expensive plant, whilst arriving at the same result in the end. Then real actual progress will have been made, and there can be no doubt but that this will soon be done.

TREATMENT OF THE "SECONDS" BOILED MASS SO AS TO OBTAIN MOLASSES FROM IT (SIMPLEX PROCESS)

The problem to be solved was the following :—It being given that with the return of the drainage liquors to the "firsts" vacuum pan and with *crystallisation in motion* there is obtained on turbinng a poor drainage liquor of 66° to 68° purity, what is the most simple and the most rapid method of obtaining in one operation the whole of the crystallisable sugar and the molasses of 55° to 58° purity?

Maguin claims to have obtained the desired result by means of—

1. A previous feeding of the "seconds" *masse cuite* by means of the "firsts" *masse cuite*.
2. Methodical cooling, regular throughout the mass, obtained in Ragot and Tourneur's exchange of temperature mixers.

Fig. 61 shows the arrangement adopted for feeding. As soon as the boiling of the "firsts" is finished and all the drainings have been

returned, the vacuum is broken in the "firsts" vacuum pan; the valve E is opened, and the vacuum being maintained in the small apparatus, the number of hectolitres required for feeding is aspirated into the latter. A gauge glass shows the number of hectolitres run in.

When the feeding is finished the valve E is closed, the vacuum is started again, and the boiling of the "firsts" goes on as usual. The drainage liquor to be boiled is then introduced into the "seconds"

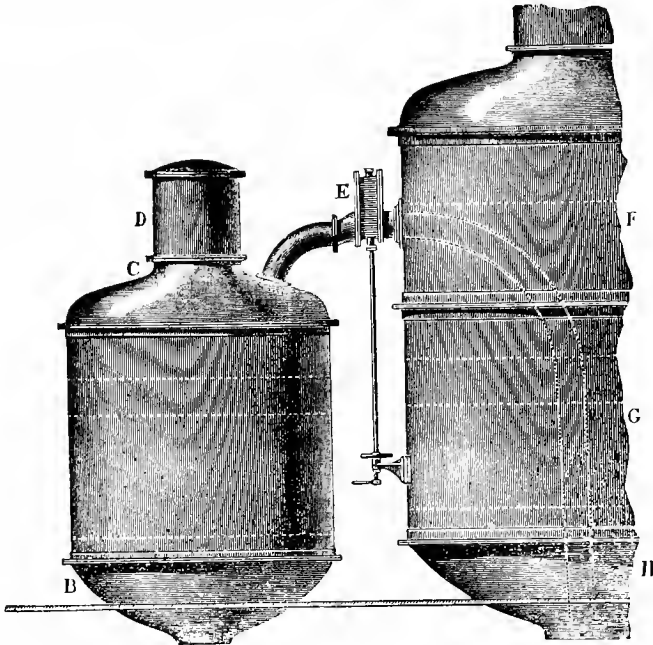


FIG. 61.—Method of feeding the "seconds" *masse cuite* with crystals from the "firsts" (MAGUIN).

apparatus slowly. It is thus exhausted of its sugar during the whole duration of the boiling by the feeding crystals previously introduced.

It will thus be seen that this operation, when well conducted, produces a rather complete exhaustion of the drainage liquor, which has now only to be aided by cooling the sugar, dissolved in the *masse cuite* in consequence of the high temperature. In fact, the mother-liquor used for feeding, drawn from the "firsts" vacuum pan, is appreciably of the same purity as the drainage liquor to be boiled, which comes from itself, and is nothing other than the mother-liquor

of a preceding boil. As soon as the boiling of the "seconds" is started, the exhaustion, which goes to increase the regular and well-divided feeding crystals, in the mass commences immediately, and is carried on during the whole duration of the boiling. If, therefore, the drainage liquor to be boiled has 68° of purity, it can easily be reduced to 63° to 65°, according to its viscosity. At this moment, the boiling being finished, the mass is cooled by means of Ragot and Tournour's cooling mixers (Fig. 62). Contrary to all other systems, in which cooling is done by a surface exposed to contact of the surrounding air or the circulation of cold water on the outside by a cold-water jacket, in the Ragot and Tournour system the cooling is done by exchange of temperatures between, on the one hand, a revolving coil in the centre of the mass, and in which a current of water circulates, and, on the other hand, the *masse cuite* itself put in

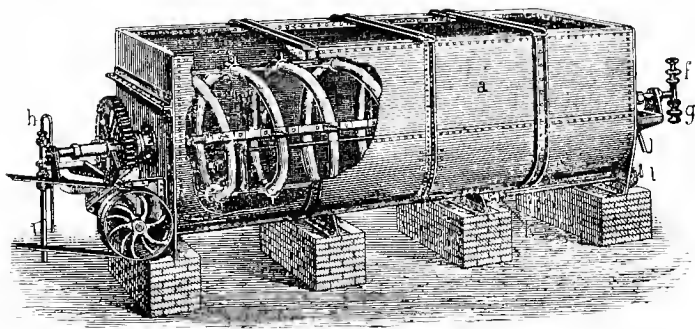


FIG. 62.—Ragot and Tournour's cooler with revolving coil, etc., in which current of cold water circulates.

motion by blades fixed on the coils. There is thus a regular and active exchange of temperature, which induces an active and uniform lowering throughout every point of the mass, and which may be made as slow and as rapid as desired by opening or closing more or less the discharge taps of the water flowing in the coil. By treatment, in the Ragot and Tournour apparatus, the temperature of the "seconds" *masse cuite* may be lowered from 80° to 85° C. to 30° to 35° C. in twenty-four to thirty hours. The exhaustion thus obtained brings the mother-liquor from 63° to 65° to 56° to 58° purity, that is to say, molasses. The *masses cuites* thus treated are easily turbinéd and yield brown sugar, saleable at 90 to 92 per cent. In certain factories where, for particular reasons, it is not desired to run the drainage back to the "firsts" vacuum pan and to produce extra sugars, the simplex system is applied to the thirds, that is to say, that the latter is fed with the crystals of the "seconds." In that case the "seconds" and

“thirds” are mixed in the cooling mixers, and a less total volume is required. The result obtained is always the same.

N.B.—Ragot and Tourneur’s mixers represent for equal volume, from the point of view of rapid cooling, three jacketed mixers and five to six simple mixers, *i.e.*, it requires three to six times less to obtain the same result. In the working of the “firsts” the cooling from 85° to 40° C. is done in six to eight hours.

RETURN ON CAPITAL INVESTED

Cost of manufacture.—The cost of manufacture in the U.S.A., says Wiley, depends on as many factors as that of beet-growing. Chief among these are transportation, fuel, weather, and labour. Perhaps the most important of these factors is the price of fuel. In some localities coal can be had for \$1.25 per ton; in others the cost may reach as high as \$10 per ton.

The manufacture of beet sugar is conducted without governmental supervision in the U.S.A., and any exact account of its cost is inaccessible. To show what it may be, with large experience and the highest skill and management, the mean cost of manufacture in 113 German factories is given:—

	£	s.	d.	\$
Mean capital invested in each factory	40,291	13	4	193,400.00
Total receipts for sugar, molasses, etc. per ton of beets	2	6	3	11.10
Mean cost of beets per ton of 2204.62 pounds	1	0	5	4.90
Salaries per ton of beets	0	1	1	.26
Labour per ton of beets	0	3	0½	.73
Interest on investment per ton of beets	0	1	6	.36
Coal per ton of beets	0	2	7½	.63
Miscellaneous expenses per ton of beets	0	4	0	.96
Total expense of manufacture per ton	1	12	8	7.84
Profit per ton of beets	0	13	7	3.26

The mean nett profit for each factory was \$34,240 (£7133, 6s. 8d., or 17¾ per cent. on invested capital). The price paid for beets, however, is in most cases fictitious, the beet-growers owning the factory, and preferring to share in the general profits rather than to charge a high price for the beets. First-class beets rarely sell for less than \$5 (£1) per ton. The Western Beet Sugar Company, of Watsonville, Cal., stated that in its first campaign, 1888–89, the cost of manufacture amounted to \$80.80 (£16, 16s. 8d.) per ton of sugar. At the present time it appears that, with the best machinery and most economical processes, beet sugar can be made in this country at a cost of from 3 to 4 cents (1½d. to 2d.) per pound when the price of rich beets does not exceed \$5 (£1) a ton.

Cost of factory.—The cost of building a first-class beet-sugar factory is much greater than is commonly supposed. From the most reliable data at hand, it may be stated that in Europe the cost of erecting a factory with the most modern machinery, of a capacity of at least 300 tons of beets per day, is about \$200,000 (£40,000). In the U.S.A. it is probable that, owing to the increased cost of transportation and the higher price of labour, the cost of a similar factory would be at least \$250,000 (£50,000). As has been intimated before, it is not advisable to attempt to manufacture beet sugar with smaller factories or with machinery and appliances which do not represent the latest improvements. It is true that there are many parts of a sugar factory which have not changed much within the last twenty years, but even the multiple-effect apparatus, the strike pans, and the centrifugals, which represent the most stable parts of the machinery, have undergone considerable changes within the time mentioned. Probably one of the greatest dangers which the beet-growing industry in the U.S.A. will meet is the tendency to begin the erection of a beet-sugar factory with cheap, old, or worn-out apparatus and appliances, and without a proper technical study of all the questions involved. The avoidance of this danger is all the more difficult because there are few engineers in this country who have devoted themselves to the study of this problem, and European experts are not likely to understand and comprehend American methods and measures. Numerous inquiries have been received by the U.S.A. Department of Agriculture for directions for making beet sugar with such appliances as a cider mill and sorghum-molasses evaporator might afford. It would not be right to encourage the attempt to manufacture beet sugar in any such way. Nor should the expectation be excited among farmers that they will be able to make a crude article of sugar which they can dispose of to a central factory for refining purposes. It is best to recognise at the very first the great expense which attends the erection of a sugar factory and the necessity for its meeting every modern requirement. Beet-growing and beet-sugar manufacture are two distinct industries, but with common aims and interests.

CHAPTER IX

THE SUGAR CANE AND ITS CULTIVATION—EXTRACTING THE JUICE BY MILLING

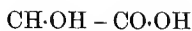
The sugar cane and its average richness in sugar.—The sugar cane is a large-sized plant belonging to the order of graminaceæ, but differentiated from the other gramineæ by the nature of its juice, which, in a suitable soil and climate adapted to its perfect growth and development, may contain as much as 18 to 20 per cent. of sugar. However, under less favourable conditions, such as are to be found, for instance, in Louisiana, U.S.A., the sugar contained in the juice does not amount to more than 15 per cent., say, in fact, much about the same as in the sugar beet.

The botanography of the sugar cane.—The structure or texture of the cane is graminaceous, the envelope is coriaceous, the interior portion consists of vessels not very close to each other, but so arranged as to form a spongy mass, in the midst of which the saccharine saps circulate, nodes at the axils of the leaves, exceedingly hard at the base, a crown of foliage bearing a flower as the plant approaches maturity. The sugar cane is furnished at each node with an "eye" capable of reproducing a new plant when it is planted in the ground. That is the way that the species is propagated or reproduced, because the cane under ordinary conditions does not bear fruit, and it is therefore impracticable to produce it from seed.

The wild cane versus the cultivated sugar cane.—The sugar cane is, in fact, a cultivated plant, not a wild one. The juice of the wild cane is much poorer in sugar. Moreover, it is altogether a much smaller plant. The wild cane reproduces itself by seeds. Hence attempts have been made lately to regenerate the sugar cane by sowing wild species. But these attempts have not as yet gone beyond the experimental stage.

Composition.—*Woody fibre cellulose and organic acids.*—The sugar cane contains not only cane sugar, but also numerous other substances dissolved in water, as well as cellulose or woody fibre. The woody fibre contains about 70 per cent. pure cellulose ($C_6H_{10}O_5$) and 30 per cent. wood gum or xylan. The following organic acids

are also present in the juice—glycollic acid, $\begin{array}{c} CH_2 - OH \\ | \\ CO - OH \end{array}$; malic acid,



|
 $\text{CH}_2 - \text{CO}\cdot\text{OH}$; succinic acid, $(\text{CO}_2 \text{H}\cdot\text{C}\cdot\text{H}_2)_2$; and small quantities

of tannic acid, $\text{C}_{14}\text{H}_{10}\text{O}_2$. Again, citric acid, $\text{C}_6\text{H}_8\text{O}_7$; tartaric acid, $\text{C}_4\text{H}_6\text{O}_6$; and aconitic acid, $\text{C}_5\text{H}_5(\text{CO}_2 \text{H})_3$, are said to be present in the normal sugar cane, but their presence has not been confirmed. Some authorities say the lime salts of these organic acids are soluble, and



FIG. 63.—The sugar cane.

(A—Encrustation of wax on epidermis, magnified. B—Floret.)

are therefore not removed by clarification, which seems rather impossible. The action of lime on the cane juice and its impurities is highly complex; it apparently gives rise to the formation of lactic acid, $\text{C}_6\text{H}_{10}\text{O}_6$; saccharinic acid, gluconic acid $\text{C}_5\text{H}_6(\text{OH})_5\text{CO}_2\text{H}$; saccharic acid, $\text{CO}\cdot\text{OH} - (\text{CH}\cdot\text{OH})_4 - \text{CO}\cdot\text{OH}$. As will be readily understood, acetic acid is only present in fermented cane.

Albumenoid and other nitrogenous substances.—The nitrogenous

substances present in the cane are principally albumenoid bodies, amides or amino acids, and xanthine bases. It has not been ascertained whether the amides or amino acids consist of asparagine, aspartic acid, or glyocol. The xanthine bases consist chiefly of guanine ($C_5H_5N_3O$), but the total nitrogen in the ripe sugar cane does not amount to more than 0.1 per cent.

Colouring principles.—The colouring principles of the sugar cane consist principally of chlorophyll and its derivatives and anthocyan, as well as a colouring principle which becomes yellow in contact with alkaline liquids.

Cane-sugar wax or cerosin.—The cane wax or cerosin which exists on the outside of the cane is said by some authorities to be soluble in alcohol, ether, chloroform, and benzine, but according to Beauvisage it is insoluble in both cold alcohol and cold ether, slightly soluble in boiling ether, but very soluble in boiling alcohol. It is obtained from the cane by scraping its stems, or, better, by purifying the skimming of the juice which has not been treated by lime. It is hard and brittle, with a clean fracture, and may be easily pulverised in a glass or marble mortar.

Moulded into a candle, it burns with a very pretty white flame. It melts at $82^{\circ} C.$ and solidifies at $80^{\circ} C.$ It may then be crystallised in interlaced truncated needles. Its density is 0.961. On cooling its alcoholic solution, it may be obtained in fine pearly lamellæ; but, on the other hand, it is said that this alcoholic solution becomes converted into an opaline mass on cooling, and that 20 centigrammes of cerosin are sufficient to solidify 30 grammes of alcohol and to give it the appearance of opodeldoc. The chemical composition of sugar-cane wax is but little known, only its elementary composition has been determined, and a formula given to it corresponding to an alcohol of the fatty series or an ether. But there is nothing to prove that it is a definite compound and not a mixture of different substances, even although a recent writer describes it as a saturated alcohol with 24 carbon atoms.

CULTURE

Reproduction not from seed but by buds.—The sugar cane, as already mentioned, is not reproduced in a similar manner to the ordinary graminaceous plants. It is, in fact, reproduced by buds.

The bad practice of using the nodes of the badly developed unripe crown as reproducing-buds.—When the cane is ripe it is cut down at a level with the ground, so as to extract the sugar from it. But, before carrying it to the mill, the crown (Fig. 63), with its dense foliage, is cut off; and it is the nodes remaining on this part of the stem which are generally used as buds for the propagation of the plant. This is a bad practice, being conducive to the degeneracy of the species. It has, in fact, been proved that the ripe portions of the cane produce

much better shoots than the nodes of the badly developed unripe crown. It is the adoption of an imperfectly understood false-economy policy.

Each plantation should have its propagation field or nursery.—It would be far better to set a field apart for propagation purposes than to use portions of the cane which are too weak to sustain the exhausting efforts of a healthy and vigorous reproduction.

Planting the buds.—The bud is planted in the ground in the bottom of parallel furrows of 6 to 8 inches in depth. They are not placed horizontally or flat in the ground, but by raising one of the ends to about 45 degrees. They are then covered with loose soil. The planting is done in moist weather, so that the germ may shoot up rapidly.

Fertile soil and a fine state of tilth absolutely necessary.—The

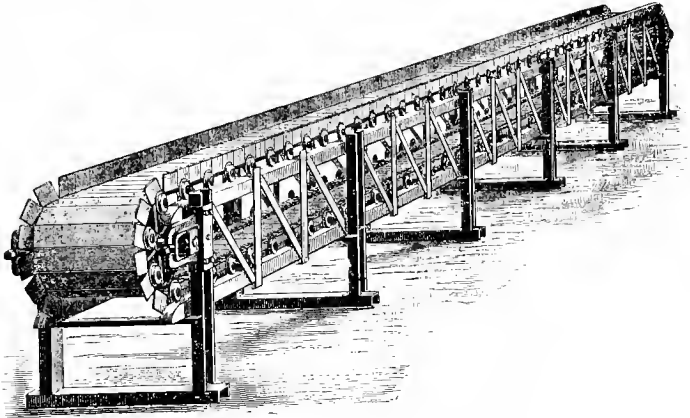


FIG. 64.—Cane conveyor (CAMBRAY).

cane requires fertile well-manured ground, but in the majority of instances the growers neglect the most elementary principles of agriculture; and the fields, in bad cultivation and preparation, and without any manure whatever, only yield but very indifferent crops. The cane requires the same state of tilth a crop of wheat requires, and the crop is proportional to the amount of care and labour bestowed upon it.

Irrigation as an aid to successful sugar-cane cultivation.—In warm climates with little or no rainfall the cane cannot sprout and shoot up without the aid of a well-planned and well-utilised system of irrigation. In Egypt, for instance, sugar can only be cultivated in certain districts by means of irrigation canals, which are fed continuously with the water of the Nile from artificial dams.

After cultivation.—After the cane is planted it soon begins to shoot forth a tuft of canes, and the fields, after several hoeings, are covered as with a dense forest, each stem being 6 to 15 feet high, and from $\frac{1}{2}$ to 2 inches in diameter.

The sugar-cane harvest.—When the sugar cane is fully developed, it is, as already mentioned, cut down on a level with the ground. The crown, with the nodes immediately underneath it, are removed, because that part, like the neck of the beet, is more impure than the remainder. The leaves are also stripped and left on the ground. The canes thus prepared are transported to the factory by the conveyor (Fig. 64) or by Bullivant's cable system. Being very bulky and cumbersome, it necessitates a large space in connection with the sugar factory in which to store it, but in any case it should be treated as soon as possible after its arrival in the factory yard.

The stubbles.—After the cane is cut, the stubbles remain in the ground. But they are still alive, and three years afterwards may still yield an abundant harvest. The fresh plants are thus cropped for three years or even longer. Consequently the planter should devote every care and attention to the fundamental portion of the work, without which abundant harvests cannot be secured. But if this initial preparation be well conducted the planter is guaranteed a long series of profitable crops. This is the reason why the discriminate selection of the plant is so necessary and fundamental a step.

COST OF SUGAR-CANE CULTIVATION AND MILLING IN CUBA

Good sugar-cane land may now (1902) be bought in every province of the island, but the best results are to be obtained in virgin soil in Santa Clara, Puerto Principe, and Santiago. As cane has to be cultivated on a large scale, and as the unit "caballeria" ($33\frac{1}{3}$ acres) is convenient when calculating on large tracts, it will be used as a unit of measurement in the following estimates. The cost of cultivating a caballeria should be about as follows:—

Clearing and ploughing	\$500, say = £100
Seed cane purchased	80 ,, 16
Planting	200 ,, 40
Cultivation	300 ,, 60
	<hr/>
	\$1080 ,, £216

$33\frac{1}{3}$ acres therefore cost £216, therefore 100 acres cost £648, or say £6, 10s. per acre, or about three times as much as it costs in Great Britain to raise a crop of wheat. If timbered land be selected and cleared, the woods therefrom should yield at least \$600, say £120 per caballeria, that is, £360 per 100 acres, or £3, 12s. per acre; about half that amount should be the initial purchase price. Consequently the end of the first year should show the land itself as a clear balance

to the credit of the planter, with a growing crop of cane representing the capital invested.

Every caballeria of virgin land should yield 1200 tons of cane to cut and haul, which would cost about \$1 per ton. This amount of cane should produce at least 120 long tons of sugar, or 268,800 pounds. A liberal estimate for milling is $\frac{1}{2}$ cent. per pound; the actual cost on a modern plantation is nearer one-fifth, or \$1344 for producing this amount of sugar. Therefore the balance sheet at the end of the first harvesting should show for every caballeria—

<i>Dr.</i>		<i>Cr.</i>
To making and harvesting crop	\$2280	Sale of 120 tons sugar @ £44, 8s.
„, milling 1200 tons cane	1344	per ton, 1 cent. per lb.
	<hr/>	\$5376
Total cost	3624	
To profit	1752	
	<hr/>	
	\$5376	<hr/>
		\$5376

\$1752 is, say £350, or £1050 per 100 acres, say £10, 16s. per acre. After the first crop is milled the total annual expense will be only about \$1500 per caballeria, thus increasing the profit to \$2532, say £500, or £1500 per 100 acres, or £15 per acre.

The authority who gives these figures says this is unquestionably a very attractive showing, but he states that it is based on thorough knowledge of the culture of cane and manufacture of sugar and careful comparison of data furnished by men actually engaged in the industry in Cuba. There is always risk of loss by fire or storm, although cane is considered about the safest from injury in that way of all crops that are produced in Cuba. However, the individual or syndicate undertaking its cultivation and conversion into sugar must remember that a large amount of capital will have to be invested in expensive plant and buildings, that the crop itself needs careful attention from planting to harvesting, and that the details of management should be entrusted only to experienced competent men.

MILLING RESULTS IN LOUISIANA

The character of the season (1888-89) producing exceedingly woody cane and a small tonnage, will account for the juices being remarkably rich that season. The first few analyses showed a high percentage of glucose. This is due to the deterioration of the cane left on the yard during the preliminary work. The delays were unusually frequent at the beginning of this season, hence considerable cane was left on the yard for several days.

The percentage of available sucrose, based upon an extraction of 72 per cent., and calculated by the formula per cent. sucrose minus one and one-half times the glucose = available sucrose, was 10.31.

The available sucrose in lbs. per ton of cane = 206·2, corresponding to approximately 214 lbs. of commercial sugar. The actual yield of sugar obtained was about 45 lbs. per ton of cane less than this amount. Not having reliable data of the mill work, it is impossible to locate the responsibility for this shortage. It is fair to presume that it was partly due to a lower extraction than 72 per cent., but this alone would not account for the shortage.

LVI.—THE YIELD OF SUGARS PER TON OF CANE, NOVEMBER 12 TO 30, 1888.

[First run, November 12 to 18 inclusive : Cane worked, 1324·5 tons ; mill work, 1217 tons ; diffusion work, 107·5 tons.]

Description.	Yield.	Sugar per Ton of Cane.
	lbs.	lbs.
First sugar (white)	116,851	88·22
Second sugar (yellow C)	63,008	47·57
Third sugar (waggon)	26,601	20·08
Fourth sugar (waggon)	15,126	11·42
Total	221,586	167·29

[Second run, November 19 to 30 inclusive : Cane worked, 1870 tons ; mill work, 1456 tons ; diffusion work, 414 tons.]

Description.	Yield.	Sugar per Ton of Cane.
	lbs.	lbs.
First sugar (white)	163,539	87·46
Second sugar (yellow C)	88,814	47·49
Second sugar (waggon)	6,833	3·12
Third sugar (waggon)	31,295	18·34
Fourth sugar (waggon)	21,355	11·42
Total	314,836	167·84

It is very probable that the class of sugars made will also account in part for the low yield. The "firsts" generally graded as "choice white." In order to obtain this grade, it was necessary to use a large quantity of water in the centrifugals. The "seconds" were graded in the pan. The molasses from "firsts" were diluted, treated with superphosphate of lime and alumina (p. 338), relimed and filtered through bone black. Again, considerable water was required to be used in the centrifugals in order to obtain a high grade of sugar.

ANALYSIS OF SUGAR CANE

1. *Sampling.*—The preparation of a fair average sample of the bulk of a lot of whole canes is really a very difficult matter. As a matter of fact, not only does the composition of different canes vary, but even that of the different internodes in one and the same plant. A fairly large number of canes, representative of the bulk, must therefore be taken and cut into rounds by means of a cane-cutter. From the cut bulk thus obtained a fresh average sample is taken, which is crushed in a mortar so as to obtain a more or less coarse pulp.

2. *Direct estimation of the sugar.*—The coarse pulp obtained in 1 is not fine enough to admit of the sugar being estimated by the cold digestion process (p. 59). The hot process (p. 59) must be adopted, and the operation conducted on twice the normal weight for 200 c.c., and so on for 400 c.c. according to the saccharimeter used. †

Process.—Add 10 per cent. of the weight of the cane of basic acetate of lead solution in c.c. Fill the flask to the mark with hot water, and digest for an hour at least in an almost boiling water bath. It is advisable to place a circular piece of metal, perforated in several places so as to prevent the pulp rising up out of the water. Cool and polarise in a 400-millimetre tube. The correction for the space or volume occupied by the mark is about 1 c.c. for 10 grammes of canes, say 3 c.c. for twice 16·29 grammes, and 5 c.c. for twice 26·048 grammes.

Exhaustion of the cane pulp by repeated washing.—Weigh out 100 grammes of cane pulp, add 200 c.c. of water and a little carbonate of lime, and bring to the boil. Draw off the solution, add 150 c.c. of fresh water, heat again, and repeat the extraction during 10 minutes, making six extractions in all, collecting the liquid in a litre flask; 8 c.c. of basic acetate of lead are added, the solution cooled, made up to the mark, filtered and polarised in a large tube, and the result calculated to per cent.

Indirect analysis.—The process for estimating the richness in sugar of cane juice is identical with that used in the case of beetroot (p. 59 *et seq.*). The cane contains about 10 per cent. of insoluble. Moreover, the first pressings, *premier jus* or *vesou*, is the richest in sugar. In order to calculate the richness of the cane in sugar from that of the juice, it is necessary to multiply by a coefficient varying between 0·87 and 0·88.

To ascertain the industrial richness of the cane, it is necessary to apply the coefficient 0·85 to the sugar of the juice in consequence of various losses and waste which the cane suffers from the time it is weighed until it comes to be treated.

EXTRACTION OF JUICE FROM CANE

The canes may be forced to deliver up their juice by two essentially different methods:—

1. The first method is by the ancient milling process, *i.e.* by expression in mills. These mills consist of a set of powerful rolls which crush the cane in its passage through them, and the liquid juice is squeezed out at the same time under the action of the strong pressure which they exert—a pressure, moreover, which also crushes the very hard nodes of the cane.

2. The second process by which the sugar cane is made to disgorge its juice is the essentially modern one of diffusion.

Sugar-cane milling.—But it will be desirable to treat of the milling process in the first instance.

In olden times the cane mills consisted simply of two vertical wooden rollers driven by a horse, the cane being passed between them. Modern engineering has, however, replaced this primitive appliance

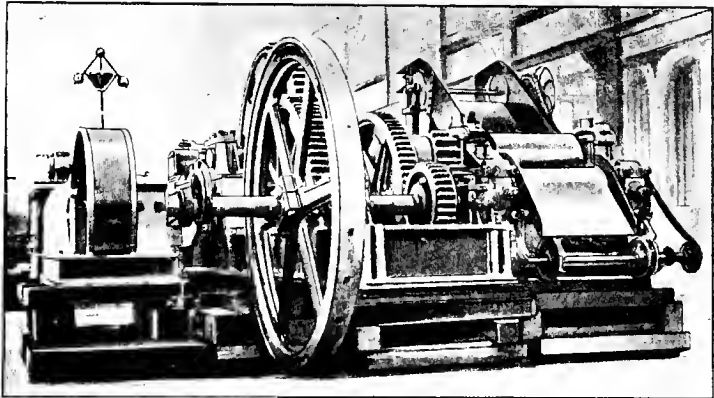


FIG. 65.—Sugar-cane mills (CAIL).

by strong mills driven by steam power, the rolls of which extract a far larger quantity of juice than the rudimentary mills of former times. Moreover, the mills have been supplemented by an arrangement by which even still less sugar is left in the cane. As a matter of fact, the centre of the cane is spongy, and every one knows that, when a sponge is pressed, the sponge again drinks up a portion of the liquid with which it was prepared to part if the pressure had been a little more strong. Accordingly the cane was made to imbibe water and pressed a second time, yielding a fresh saccharine liquid, more dilute, it is true, but none the less profitable.

Cane shredders.—They have even gone a step further. Machines have been invented, called shredders, which tear the cane up into fibres more easily moistened and pressed, and yielding a still larger quantity of juice.

It is thus possible to extract as much as 90 per cent. of the juice in the cane, whilst the mill by itself alone can only express some 70 to 75 per cent.

Description of a modern horizontal sugar-cane mill.—A sugar

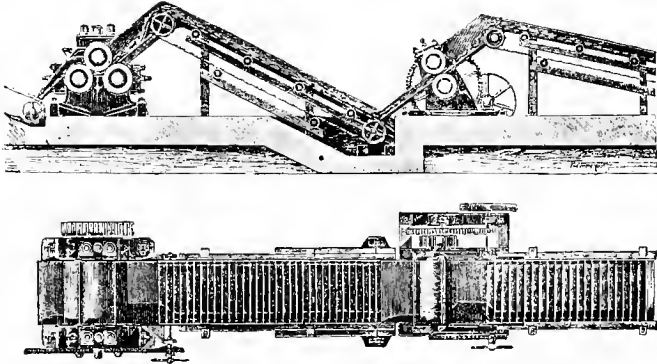


FIG. 66.—Milling followed by immersion and subsequent remilling (CAM).

mill consists of three horizontal cast-iron cylinders or rolls arranged in the following way. The axes of two of the rolls (A and B, Fig. 68) are in the same horizontal plane, and are quite near to each other without touching, yet are sufficiently far apart to leave between them

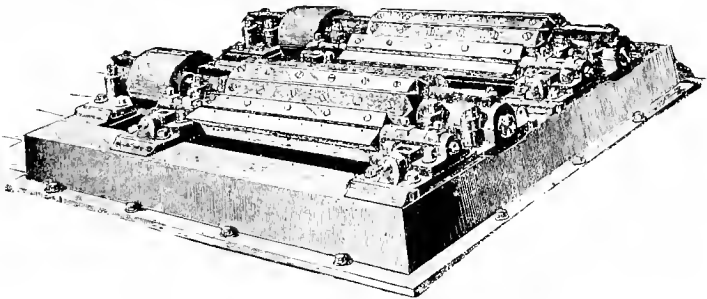


FIG. 67.—Cane shredder (CAMBRAY).

a space, varying with the size of the mill, between 8 and 10 inches. The third cylinder (C) is superimposed on the two others in such a way as to touch them both.

The axes of the two cylinders (Figs. 66 and 68) are held in position at their extremities in stuffing-boxes in a very strong cast-iron frame. The distance between the rolls may be varied by pressure screws.

Strong pinions render the three rolls firm and solid in such a way that they turn together, acting as one only—the top one. Gearing and shafting (Fig. 65) connected with the axis of the top roll are driven by a steam engine, in such a way as to impart to the rolls the necessary and suitable speed.

The cane is brought, by an inclined plane (Fig. 68, D), between the top roll and one of the bottom ones. The rotation motion brings it under the top roll, and from there it passes between the same roll and

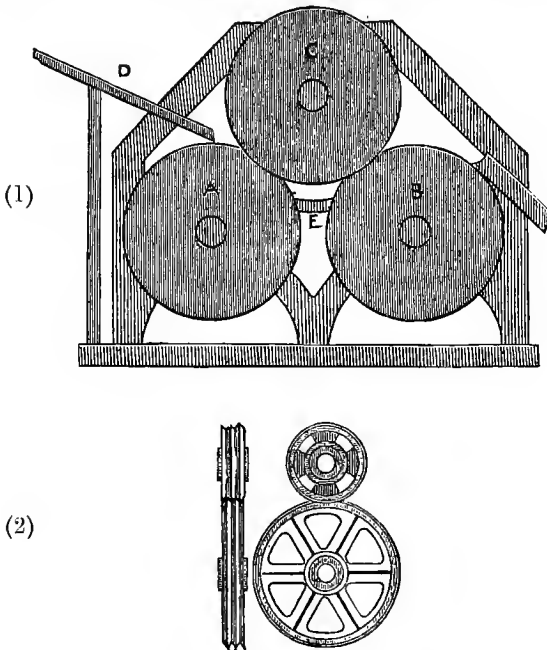


FIG. 68.—Diagrammatic representation of the cane mill, showing (1) rolls and (2) friction wheels.

the second lower roll, being led in that direction by a guide plate E placed between the two lower rolls. Finally, it makes its exit, and falls on an inclined table, and afterwards on a conveyor which carries it outside the factory.

To carry the cane to the mill, a long mechanical conveyor (Fig. 64) rolls in front of the workpeople, who throw the canes into it by armfuls, and it thus arrives in front of the inclined plane (Fig. 68) which feeds the mill.

The cane conveyor and the bagasse conveyor are driven by the

mill itself in such a way that, when it stops, the feeding of the mill stops.

Size of mills.—The sizes of mills are very variable. Some are very small. But ordinary big mills have rolls 30 inches in diameter and 78 inches long, crushing 250 to 300 tons of cane in 24 hours. The rolls make two revolutions a minute.

A diagrammatic representation of the mechanical principles of the three-roll horizontal cane mill is illustrated in Fig. 68. A, B, C are three cast-iron rolls with flanges at their ends to prevent the escape of the canes. In the case of large factories these rollers are very massive and ponderous, attaining the dimensions 6 feet in length by $2\frac{1}{2}$ feet in diameter. The upper roller C is occasionally fluted longitudinally so as to get a good firm grip of the cane, and thus make sure of drawing it into the mill. Nevertheless, these longitudinal grooves are not indispensable. Moreover, as the grooves hash up and crush the cane very roughly and otherwise injure it, the smooth uniform surfaced roll is preferred. The three rollers revolve simultaneously, being driven by means of cog-wheel gearing. Either steam, wind, water, or animal power may be used.

D is an inclined table down which the canes descend to reach the first set of rolls A, C. These rolls are about quarter of an inch apart. The distance may be regulated by screws. The cane is well crushed between the first set of rolls A and C, and converted into a fit condition to benefit by the extreme pressure which is afterwards imparted to it in its passage between the rolls B and C.

At E is a guide plate upon which the crushed canes are deflected upwards, after passing the first set of rolls, so as to cause them to pass between the rolls C and B. These rolls are much closer together than A and C—so close, in fact, that a sheet of paper can hardly be passed between the two. Under this energetic pressure the juice is squeezed or expressed from the cane, and drips from the rolls into a gutter, which conveys it to vessels where its further treatment is begun.

It will be readily understood that the rolls lying so near to each other will be more or less liable to get jammed when irregularly fed with too bulky a charge of cane. A sudden stoppage of this kind would inevitably lead to a serious break-down,—either the rollers themselves would be injured, or the cog-wheel gearing or shafting by which they are driven. Various measures are adopted to avoid this calamity. The rolls, for instance, may be driven by friction wheels, the nature of which will be readily perceived from the accompanying illustration (see lower portion of Fig. 68).

These, instead of being on the tooth or cog-wheel principle, have a series of ridges on their circumference which fit into a corresponding series of ridges. When a pair of wheels constructed in this way are pressed together the friction thus produced causes the revolution

of the one wheel to make the other wheel revolve also. But if the wheel which is being driven in this way be stopped, or encounter some very formidable resistance, no break-down whatever occurs, as the driving wheel merely glides or slips upon the other without causing it to revolve. The friction between the two wheels must be rather greater than that required to work the cane mill under normal circumstances.

Break-downs to the mill machinery caused by the jamming of the rolls may be avoided by an arrangement depending upon quite a different principle entirely. Under ordinary conditions the distance between the rolls is adjusted by screws, but it will be at once seen that if one of the rolls could be pushed towards the other with the requisite force there would be no need for screws. This is what is done in the case of the roller B, which is pressed towards the roller C by great pressure generated by levers and weights. Any very unusual strain upon the rollers is, however, sufficient to overcome this pressure which urges B towards C. When this takes place, therefore, the roll B is pushed back from C, and the obstacle causing the obstruction is thus allowed to pass through.

Influence of speed of rolls on percentage yield of juice.—It has been found in actual practice that a larger percentage yield of juice is extracted from the cane when the speed of the rolls is about two to three revolutions a minute than when driven at a greater speed.

Looking to the vast mechanical power brought to bear upon the cane in its passage through the sugar mill, it might at first sight be imagined that the great bulk of its percentage of juice would be expressed. Although great improvement has no doubt taken place of late years, yet not so very long ago not more than 50 per cent. of the juice was extracted on an average. The causes of such a deficiency were many and varied: some of these were due to inherent defects in the construction of the mills themselves, others to imperfect handling, whilst still others were due to sheer prejudice.

The good or bad working—that is, the greater or less efficiency of a mill for crushing canes—will obviously depend, other things being equal, on the distance between the rolls. As the effect of the wear and tear of continued working, the distance between the rolls naturally tends to increase; and notwithstanding that this distance may be regulated by screws and bearings, so that the rolls may be brought as near to each other as may be necessary, yet this operation involved so much time and trouble that it was very often neglected.

The great point to be attended to in maintaining the efficiency of a sugar mill is in seeing to the regularity with which it is fed. The main point, in fact, to be always borne in mind is that the canes should be fed into the mill in an even, uniform layer. If this point does not receive the attention its importance deserves, then the canes, being fed into the mill unevenly, will be unevenly and irregularly

pressed. The canes in the thick portion of the layer will receive great, if not undue, pressure, whilst the thin edges, if they do not escape pressure altogether, will at least not be pressed to the extent they would be had the mill been fed in a rational manner.

The point now insisted upon was one of the great objections to the old-fashioned vertical rolls. These vertical cylinders had to be fed much more carefully than the horizontal rolls.

But, however excellent the sugar mill may be, and however perfect may be its working condition, and however carefully and punctiliously it may be fed, one great cause of imperfection still remains. This is due to inherent defects in the mechanical principles on which the sugar mill is constructed.

From very obvious mathematical deductions it will be readily granted that the line of maximum pressure between the two proximate revolving rolls (cylinders) is infinitely small in width. It naturally follows, therefore, that the width of the layer of cane at one moment subjected to this pressure must be infinitesimally small. However, if the woody fibres and cellular tissue composing the sugar cane were altogether deprived of elasticity, there would be but little force in this line of argument. But, as a matter of fact, the cane is comparatively rather elastic; it, as soon as it passes over the line of maximum pressure, expands, whilst at the same time this expanded portion acts exactly like a sponge and again sucks up a portion of the juice as soon as it has been expressed. This was a defect inherent to the principle of construction of the mill, which could not of course be altered so long as that principle was adhered to.

Bessemer's machine for expressing the juice from the sugar cane.—

With the view of obviating this defect inherent to roller mills, and with the object of more completely extracting the juice from the cane than is possible in any mill built on the roller principle, Sir Henry Bessemer invented a machine for expressing juice from sugar canes by means of plane contact—that is, by the pressure of two flat surfaces like a hammer on an anvil. This machine when in action performs two separate functions: it not only cuts the cane into short lengths, but squeezes them at the same time.

A diagram of the principle on which this mill works is shown in Fig. 69. P is a solid plunger which oscillates to and fro in a tube. The plunger is driven by a crank, and receives considerable power with the aid of the inertia of a fly-wheel. The canes are supplied to the mill by the vertical tubes A and B. The plunger in the position shown in the figure is about to move towards D. In its passage it cuts off the end of the cane in the tube B, and compresses the segment thus cut off against the mass of bruised cane D. The juice which flows from the cane escapes through the holes H H in the tube. These holes are conical, with the narrow end inside to avoid clogging. On the return of the plunger to C a segment from the end

of the cane in A is cut off, and has its juice expressed. A new length of cane in B descends into the tube to be ready for a fresh operation.

Thus two canes are supplied to each tube at the same time, and several plungers and tubes can be worked by the same engine. It is believed that by this process the juice is more completely expressed than by rolling. Not only is sugar saved, but the expressed canes (bagasse) are drier, and therefore more fit for fuel; and this is a very important consideration where fuel is scarce and expensive.

Exhaustion of the bagasse.—The bagasse as it comes from the mill still contains 25 to 30 per cent. of juice. As already stated, it is drenched with water and crushed again. With this end in view, the bagasse conveyor passes through the bottom of a flat tank filled with water, and brings up the moistened bagasse to pass under a new mill, consisting often of only two superimposed rolls (Fig. 66), but some-

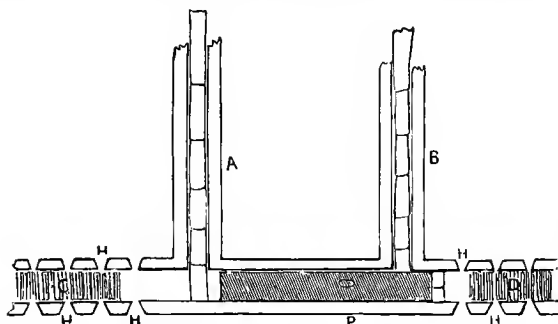


FIG. 69.—Diagrammatic representation of Bessemer's machine for forcing the juice from the sugar cane.

times under a similar mill to the first. The bagasse is moistened and milled twice so as to leave as little sugar as possible.

Shredders.—The shredder which is sometimes used—*Faure's* shredder, for instance (Fig. 67)—is a mill with grooved rolls turning at different speeds. The cane is passed up under its efforts in an inverse direction, and is more easily pressed and moistened.

A shredder invented by *Baze* consists of a steel arm passing between the bars of a strong grating in such a way that the cane falling underneath its arm is hashed up into small pieces, whilst still preserving the form of fibres long enough to yield a good pressing and capable of being easily moistened.

SYSTEMATIC WASHING OF THE BAGASSE—PERICHON'S PROCESS

This process is adopted in the factory of Rodah, Egypt, belonging to the Daira Sanieh of the Khedive. Its object is to extract the sugar

left in the bagasse produced by the passage of the cane, either through a single mill or through two consecutive mills without imbibition between the two crushings.

The bagasse thus obtained after one or two crushings falls on a conveyor, which carries it into the washing tanks, mounted on wheels and so arranged that the liquid which they contain can flow out as quickly as possible and the exhausted bagasse can be discharged without difficulty.

For this purpose, each washer has a wide discharge valve in its

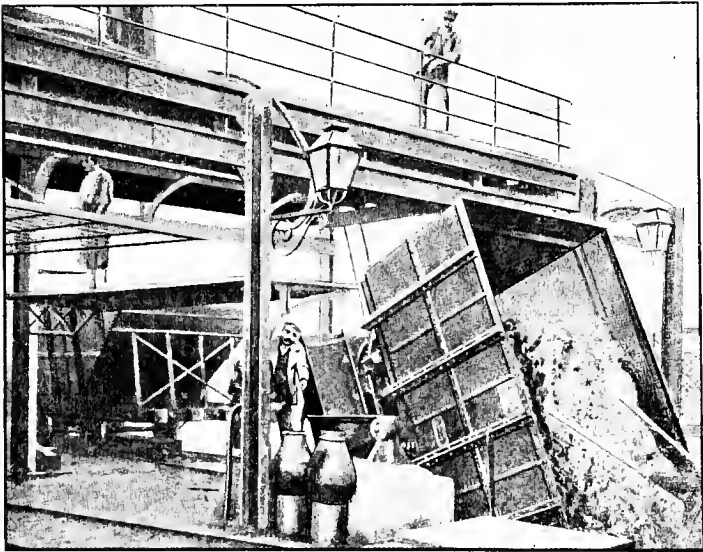


FIG. 70.—Systematic levigation of the bagasse. Perichon's process, as used in Egypt (C.A.I.L.).

lower part under a perforated wrought-iron false bottom, and at a convenient height, a movable screen, which is removed when filling in or discharging the bagasse, and which is fixed to the sides of the tank to keep the bagasse at a uniform height during the systematic exhaustion.

The wash-tanks containing the exhausted bagasse are run in succession on to a hydraulic bascule, and after removing the movable screen they are tilted (Fig. 70) on to a conveyor, which carries the bagasse to a crushing mill intended to remove the greater part of the water, so as to enable it to be afterwards used as fuel.

Hot water is run into the battery of wash-tank tilting waggons, the number of which depends on the density desired in the wash-liquors, the nature and richness of the bagasse, and the exhaustion which it is desired to attain, in the wash-tank whose bagasse is most exhausted. The wash-water, as will be explained further on, passes in succession through the wash-tank tilting waggons, and becomes richer in sugar as it passes from the one to the other, until it reaches the last, filled with fresh bagasse as it comes from the mills, so as to form the wash-juice, which is sent to the factory to be treated either by itself or in admixture with natural cane juice.

The battery being in full working order, when the time comes to remove the wash-tank tilting waggon containing the exhausted bagasse, the liquid in which the bagasse is immersed in the different tank waggons is previously and simultaneously emptied and pumped into as many calorisors on an upper floor as there are wash-tanks in the battery.

The entire battery is then shifted back by the length of the suppressed wash-tank in the rear, and a new tank charged with fresh bagasse from the mills is run on in front. Then the wash-liquors previously removed and reheated in their respective calorisors, where they have been methodically treated with lime, are run back into all the wash-tanks of the battery.

These dischargings, shiftings, and filling of the battery are repeated each time that a wash-tank containing exhausted bagasse is withdrawn from the front of the battery and replaced behind by another charged with fresh bagasse.

Perichon's process, as described above, has been used during three seasons in the Rodah factory. It consists of two batteries each of eleven wash-tank tilting waggons for treating the bagasse coming from two successive crushings without imbibition between the two pressings; and it gives every satisfaction, both by simplicity in working and by the results obtained, which are much superior to those previously obtained in the same factory by re-pressing after imbibition. In fact, in Rodah factory, which in 1900-1 treated 1160 tons of cane per twenty-four hours for ninety-six days, there was obtained a total extraction of mixed natural juice and washed juice of 89.53 litres only per 100 kilogrammes of cane, and the average density of the wash-juice at 15° C. was 1.043 (6.2 Baumé), whilst this average of the natural juice at 15° C. did not exceed 1.068 (9.45 Baumé).

The yield was—

	Per cent. of Cane.
Sugar No. 1, polarising 98.50	10.17
Sugars Nos. 2 and 3, which were not yet turbinéd, polarising about 84 (estimation)	0.60
Final molasses, polarising about 34 (estimation)	2.80

The average richness of the cane was 12·2 per cent. of sugar, and the quantity of sugar left in the exhausted bagasse was about 0·4 per cent. of the cane treated.

N.B.—This process may be used with fixed batteries as well as movable batteries like those of Rodah.

The Daira Sanieh, satisfied with the results obtained at Rodah, decided to use this process in the season of 1901–2 in his Bibeh factory, where the bagasse from 1800 tons cane would be treated daily.

Increase in the density of the wash-juice from the different wash-tanks which compose the two batteries installed in the Rodah factory:—

Left Battery.		Right Battery.	
No. of Wash Tank.	Density at 15° C. ° Baumé.	No. of Wash Tank.	Density at 15° C. ° Baumé.
1 exhausted bagasse	0·4	1 exhausted bagasse	0·45
2	0·8	2	1·0
3	1·3	3	1·6
4	1·9	4	2·0
5	2·7	5	2·7
6	3·3	6	3·3
7	4·0	7	3·9
8	4·6	8	4·7
9	5·4	9	5·6
10	6·2	10	6·1
11 final withdrawal of juice	6·3	11 final withdrawal of juice	6·3

BAGASSE AS FUEL

Its use as fuel.—The final bagasse is used to stoke or fire the boilers, in which it burns like straw when it is dry. But this drying is not always easily accomplished. Moreover, the rind of the sugar cane, like that of most grasses, contains much silica, so that when it is burnt a glassy slag results which renders the crushed cane ill adapted to serve as fuel.

In very hot countries with no rainfall it suffices to spread it on a field, and the sun soon dries it. But in countries like Louisiana, where the climate is a rainy one and the sun sometimes not to be seen, the bagasse has to be stored in sheds, which is a dangerous practice on account of the heat generated by the fermentation being liable to cause conflagrations. Moreover, it is very costly, seeing the enormous volume occupied by the bagasse from a few days' working. If this be not done the bagasse has to be burnt in the wet condition, after having been more or less energetically pressed in the last mill. But this wet bagasse does not burn unless it be dried in the furnace itself, where it is by special arrangements exposed to the heat of the flame before being burnt. But such a fuel produces but little steam. Nevertheless, as it is the only way of getting quit of it, this has to be

put up with, and the small number of calories generated by it in the boilers taken as much advantage of as possible.

The furnaces for burning wet bagasse are numerous; but, as will be seen later on, diffusion bagasse is altogether different.

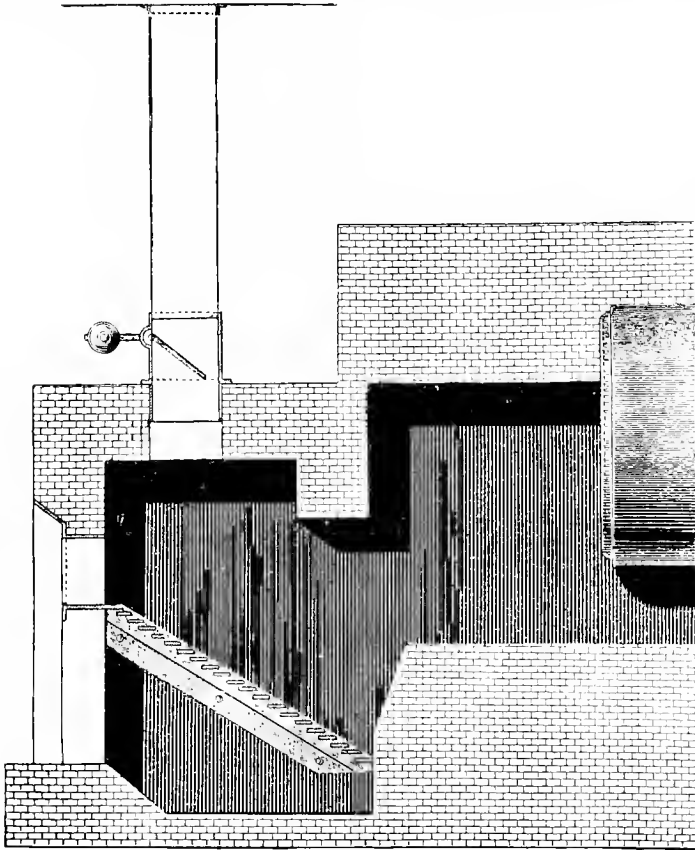


FIG. 71.—Furnace for burning green bagasse (CAMBRAY).

Furnace for burning green bagasse.—The accompanying illustration (Fig. 71) shows a furnace for burning green bagasse as it comes from the mills, with an inclined grate or hearth. Sometimes an oscillating grate is used, so as to let the ashes fall through as soon as formed, thus reducing hand labour.

CHAPTER X

SUGAR - CANE DIFFUSION — THE RESULTS OBTAINED ON THE LARGE SCALE BY THE UNITED STATES DEPARTMENT OF AGRICULTURE AT MAGNOLIA, LOUISIANA, U.S.A.

THE principle of diffusion was applied to the extraction of sugar from the sugar cane as a natural sequence to the success obtained with the sugar beet (Chap. IV.).

A cane-sugar diffusion battery is altogether similar to a beet diffusion one. The only difference is in the shape of the diffusers (Fig. 72), which should be truncated, the largest base underneath. For evacuation purposes, the discharge door occupies the whole of the bottom.

The diffusers are heated to a much higher temperature than the European beet diffusers (p. 315), and the diffusion process is carried on under altogether favourable conditions, because it is not really a diffusion process,—it is simply a washing of the slices, which immediately part with their saccharine juice.

THE MAGNOLIA DIFFUSION EXPERIMENTS

The results of the diffusion work at Magnolia, Louisiana, U.S.A., though unsatisfactory in some respects, thoroughly demonstrate the practical manufacturing value of the process as applied to sugar cane. The cane, says Mr. Spencer, will submit to rougher treatment in the diffusion battery than the beet, and consequently the manipulations are simpler. This very property of the cane often tempts the battery-man to careless work, resulting in loss to the planter. Every possible precaution should be taken to secure regularity of work. It should be remembered that the battery-man is placed in a responsible position, and he should be remunerated accordingly. Delays incident to the diffusion battery were of rare occurrence. With a satisfactory cane-cutter there is little probability of delays, except from bad weather. The results of 1888 season's work indicate the possibilities of diffusion, and justify a rapid introduction of the process.

The milling of exhausted chips for use as fuel.—On page 316

attention is called to the probable effect of high temperatures on the cane, especially in regard to the subsequent milling of the exhausted chips. In 1887 the milling experiment was practically a failure; whereas in 1888, on the contrary, it was successful. No special adjustment of the mill was made for the experiments in either case.

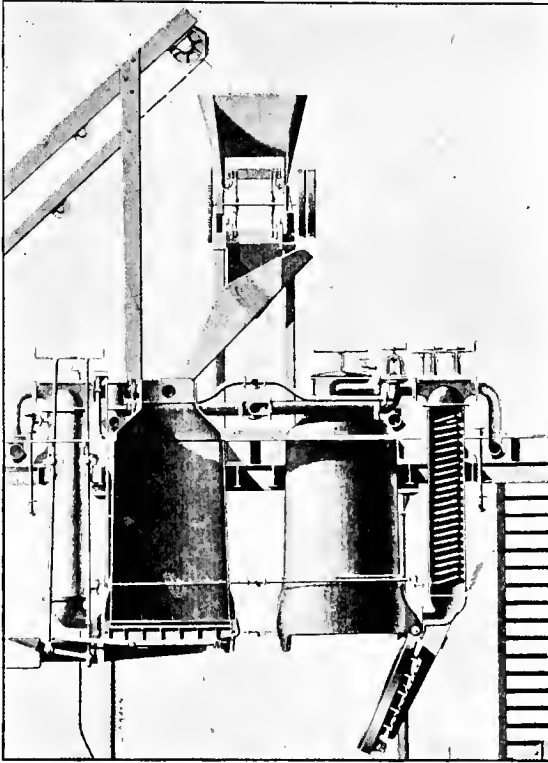


FIG. 72. — Cane-sugar diffusers, showing elevator, feed-chute, and caloriser, etc. (C&L).

The following percentages of water were left in average samples from each mill in 1888 :—

- | | | | | | |
|-----|------------------------------|-------------|-------|-----------|--------|
| (1) | Chips from three-roller mill | retained | 60·85 | per cent. | water. |
| (2) | „ | five-roller | 52·65 | „ | „ |

The chips burned freely; those from the three-roller mill would probably have burned fairly well, but no test could be made.

The milling and burning of the chips is a less difficult problem than it is usually considered. It would certainly be much more economical than the present practice at Magnolia of dumping them into the Mississippi River.

Coal consumption in sugar-cane diffusion working.—The value of the figures which Mr. Spencer obtained in his diffusion work at Magnolia was very much lessened by the adverse conditions under which steam was generated in the bagasse boilers. The bagasse chute (Fig. 71) was arched over with brick, and coal was used for firing. It is evident that economical steaming was out of the question under these conditions.

The bagasse burner was built under the Fiske patents. The boilers were cylindrical. In the rest of the steam plant the boilers were of the double-flue type, designed for burning coal. The figures are not given as an exhibit of economical management, but are simply a statement of actual work.

The total consumption for the diffusion work was 2,674,585 lbs. This includes all the coal used, except in bone-black room.

The fuel burned in swing-out sugars after the close of the season is divided between the mill and the diffusion work in proportion to the yield of sugar. The only basis for estimating the coal consumption for each run is the relative actual dilution (see p. 317).

LVII.—RÉSUMÉ SHOWING THE COAL CONSUMPTION FOR EACH RUN.

Period.	Coal consumed per 1000 lbs. Sugar.	
	Lbs.	Barrels of 180 lbs.
Third run	1942	10·79
Fourth run	2029	11·27
Fifth run	1757	9·76
Average for the season	1875	10·42

In examining the coal statement, work must be taken into account. In general it is safe to estimate very nearly as large a fuel consumption during irregular work as when the house is working to its full average capacity. Since the introduction of diffusion at Magnolia the coal bills have been more than doubled. Taking into account the numerous delays and the failure of the cutter to furnish sufficiently thin chips for work at a low dilution, a lower consumption of coal could hardly have been reasonably anticipated.

It is conservative to estimate a coal consumption of from 1200 to 1400 lbs. per 1000 lbs. of sugar as sufficient under favourable conditions. In this estimate no account is taken of the exhausted chips, which ought to furnish a large proportion of the fuel.

DIFFICULTIES ENCOUNTERED IN SUGAR-CANE DIFFUSION

The great difficulty in sugar-cane diffusion work is *to find* a cane-cutter. That which is generally used consists of a revolving plate like that used for beets, but the knives are simple plane-cutting blades. Above the revolving plate are six peculiarly shaped hoppers, fixed in such a way that the cane fed into them presents itself in front of the knife under a certain angle. The cane is cut on the slant, and not perpendicular to the axis. Rounded, elongated slices are thus obtained, which fall into the diffusers from a chute in the same way as in the case of beets.

DESCRIPTION OF CANE-CUTTER CONSTRUCTED BY THE SANGER-HAUSEN COMPANY OF SANGERHAUSEN, GERMANY

This cutter consists essentially of a horizontal disc carrying twelve knives set parallel to the radii of the disc, and revolving in a cast-iron shell. There are six cane chutes or hoppers placed at an angle of 45 degrees to the surface of the disc. The cane, thrown lengthwise into the chute, descends by gravity to the knives, where it is sliced diagonally. A suitable arrangement for throwing the chips into the elevator boot is provided. This cutter was designed by the constructors to revolve 110 revolutions per minute, and its capacity was guaranteed to be from 200 to 250 tons of chips per twenty-four hours. After overcoming numerous and serious faults in the construction of the cutter and increasing its speed to 180 revolutions (an increase of 63 per cent.), 195 tons of chips were cut from straight cane in twenty-four hours, the largest day's work accomplished at Magnolia.

An able and experienced sugar manufacturer had charge of this work, and it was possible by his skill to overcome the difficulties in preparing the cane for the battery. After the first failure of the cutter, Dr. Wiley ordered small steel scrapers to be attached to the upper surface and side edge of the cutting disc. The cane was very juicy in 1887, hence he was enabled to cut nearly 1000 tons of cane without appreciable wear of these scrapers. In 1888, on the contrary, the cane was exceptionally woody, and scrapers of the best file-steel were worn out in cutting less than 400 tons of cane. In addition to the trouble with these scrapers, it was found that the fibres from the cane collected between the disc and outer shell and soon completely blockaded the cutter. Openings were cut both in the cover and upper part of the shell to relieve the disc of the accumula-

tions of fibre. It was only after these alterations were made that it was possible to use the cutter at all.

Economical diffusion of cane sugar demands an exceedingly thin

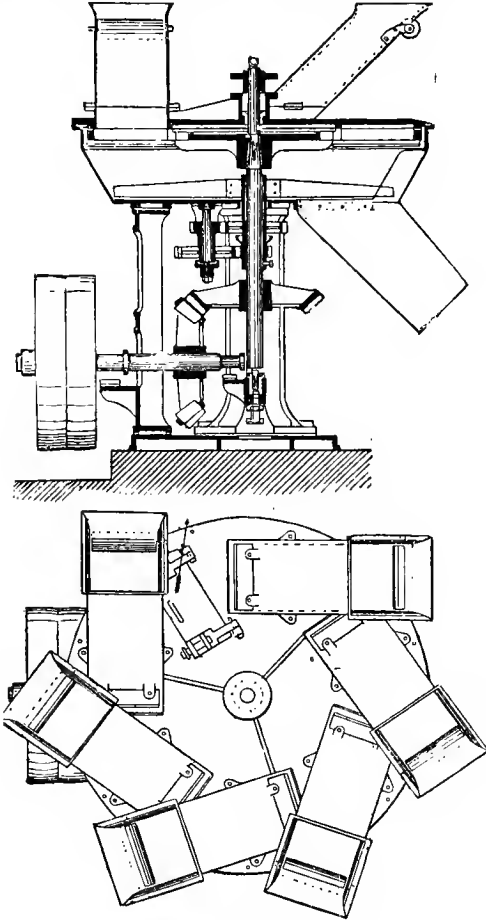


FIG. 73A.—Cane cutter (C.A.I.L.).

slice or chip. With the best possible working at Magnolia it was impracticable to obtain a chip less than an eighth of an inch. This is double the thickness required by good work.

Taking into consideration the large labour bills, the difficulty in

regular adjustment of the knives, and impossibility of obtaining a sufficiently thin chip, this cutter, concludes Mr. Spencer, is not suitable for diffusion work in Louisiana.

The Hughes cane-cutter.—The system of cutting cane used at Colonel Cunningham's estate in Texas, and in the sorghum houses in Kansas, is the invention of H. A. Hughes of Cape May City, N.J. This cutter consists of a metallic cylinder, carrying a number of knives whose blades project from the circumference of the cylinder in the direction of rotation. The cylinder is rapidly revolved in front of a dead knife set parallel to its face. The cane, previously cut into short pieces, is thrown into a hopper, where it is caught by the knives and carried against the dead knife. A small piece of cane is cut off and carried between the knife and dead knife, and by the centrifugal force is thrown into a receiver below. This cutter or shredder is always used in connection with an ensilage cutter, which latter furnishes the short pieces of cane.

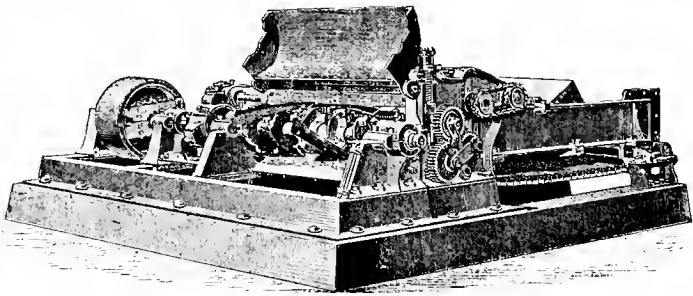


FIG. 73B.—Cane-cutter.

The rapid advancement of cane diffusion is largely due to Mr. Hughes' successful cutting apparatus.

The U.S.A. national cane shredder.—Many visitors at Magnolia plantation, Mr. Spencer reported, suggested the adaptation of the cane shredder to the preparation of cane for the battery. This machine was used several seasons by Governor Warnoth in the shredding of whole cane for the mill at Magnolia. If its work during this time can be taken as a criterion, the shredder could, in Mr. Spencer's opinion, be readily adapted to the requirements of diffusion.

DETAILS OF SUGAR-CANE DIFFUSION WORK

The diffusion battery.—Mr. Spencer gives the following details of the diffusion battery used in his experiments on the large scale at Magnolia plantation. It was built in 1887 by the Colwell Iron

Works of New York. It was enlarged in 1888 by Edwards & Hauptmann of New Orleans, according to the directions of Governor H. C. Warmoth.

The number of cells in the battery.—In the enlargement of the battery the only changes made were in the length of the cells and calorisors or heaters, and the addition of two new cells. The battery as used consisted of 14 cells, arranged in a circle, and charged from a central reservoir by means of a revolving chute (see Fig. 72, p. 302).

Dimensions of the cells.—The cells were 11 feet long by 44 inches in diameter; the nett cane space 107 cubic feet; the upper doors 30 inches in diameter, and the nett opening at the discharge door 44 inches, the full diameter of a cross section of the cell. The joint between the discharge door and the bottom of the cell was the ordinary hydraulic closure (p. 97).

Calorisors.—The calorisors or heaters (Fig. 72), as originally constructed, were 49 inches long by 2 inches in diameter in each, giving a heating surface of 17.1 square feet. In enlarging the battery, seven tubes, $1\frac{1}{2}$ inches by 41 inches, were added, giving an additional heating surface of 9.2 square feet per calorisor. The heating surface was sufficient for the work, but it would have been a wise precaution to have increased it considerably more. The heating surface per cubic foot of cell space was 0.246 square feet, or nearly 4 cubic feet per square foot of heating surface.

Juice and water mains, etc.—The juice and water pipes were of cast iron, and have a nett diameter of 4 inches. The compressed air pipes were $1\frac{1}{2}$ inches in diameter. The accumulator for compressed air had 75 cubic feet capacity. A 2-inch main furnished ample steam for the battery.

Method of removing exhausted chips.—A circular track under the cells, provided with a flat car having its axles fixed in the direction of the radii of the circle, served to carry the chip car from cell to cell. The flat car was fitted with a piece of track of the same gauge as that of the permanent railroad leading to the river. When a cell of exhausted chips was discharged into the car, the flat car was drawn by a mule to a point opposite the main line and the chip car run off and taken to the river to be emptied. The round trip required less than seven minutes. A large flat-boat projecting into the river served to carry the track far enough out for the current to wash the chips away.

Circular batteries versus line batteries.—The question of arrangement of a diffusion battery will generally depend upon local conditions. The batteries built for the U.S.A. Agricultural Department previous to that at Magnolia were of the type known as line batteries. The circular arrangement was selected for Magnolia in order to give the planters an additional example of the different types of diffusion

batteries. The circular arrangement has many advantages. It also has disadvantages with which the line battery is not compelled to contend. The principal of these latter is the difficulty attendant upon the removal of the exhausted chips. A builder of this class of machinery informed Mr. Spencer that there is no difficulty in arranging to move the chip car from cell to cell by power and finally run it outside the building for dumping.

A circular battery possesses decided advantages over all other forms in ease and regularity of charging the cells with cane chips, neatness of arrangement, and facility of controlling the work. The valves should be so arranged that they can be manipulated from inside the circle. The measuring tank should also be placed inside the circle, preferably at the centre.

Defects in the Magnolia battery.—The defects in the battery were not due to the workmanship, but rather to the designers and to oversights when increasing its capacity. The department is in no respect responsible for these latter.

The chip chute.—The chute (Fig. 72) should be entirely supported from above, a counterpoise relieving the strain caused by the weight of the chute coming entirely on one side. Instead of a sliding door, to block the flow of chips when moving from one cell to another, the end of the chute should be provided with a hinged spout, balanced in such a manner that it can be thrown back and stop the flow of chips, the bottom of the spout becoming a gate. When this arrangement is adopted, there is ample room to place the measuring tank in the centre of the upper platform. The valves and pipe-lines being on the inside of the circle, the battery-man has easy control of the work, and cannot be pardoned for overheating the cells or making other errors.

In the enlargement of the battery the size of the pipe-lines was not proportionately increased. We found for rapid work—*i.e.* a cell every seven and one-half minutes—that a pipe area of $12\frac{1}{2}$ inches is not quite sufficient, but 20 inches would be ample for a cell of the dimensions of those at Magnolia.

The calorimeters or heaters were of sufficient capacity. In enlarging the calorimeters the original outlets for water of condensation were retained.

Control of diffusion work.—In order to arrive at comparable results, and place the records beyond the possibility of error through neglect or forgetfulness of the workmen, some automatic device for registration is essential. A number of German and other beet-sugar manufacturers have devised instruments for recording all that is essential in the work of a battery. These records are the work of an instrument, and, made entirely without prejudice or fears of punishment for negligence, become valuable data for locating and correcting errors. The first cost of such instruments will be many times repaid. A battery-man, no matter how faithful and capable he may be, is

liable to make errors that may prove very expensive, and render valueless studies to improve the work.

The diffusion of sugar cane presents many conditions quite different from those which exist in the beet.—The comparatively small amount of work that has been done in the diffusion of cane in the U.S.A., and many of the conditions under which it has been done, have prevented a careful study to determine the most favourable conditions for such work. Now that the success of the process is fully demonstrated, we should turn our attention to improving the work of our batteries. In addition to the automatic records of Horsin-Deon's and Eugene Langen's apparatus (see Fig. 31, p. 133, and Fig. 33, p. 135), the following further checks on the battery work were adopted. All the automatic registers were kept under lock and key, and out of sight of the battery-man. This workman was provided with a checking system that promptly notified him of errors.

For this purpose, blanks, ruled as below, were furnished the men at Magnolia.

LVIII.

MAGNOLIA PLANTATION.									
Date.....					Watch.....				
Battery-man.....									
Cell No.	Time when Drawn.	Density.	Temperature.	Litres Drawn.	Cell No.	Time when Drawn.	Density.	Temperature.	Litres Drawn.

The men were required to fill in the blanks and enter on the back of each sheet the cause of delays.

The most frequent error is drawing two or three times from one cell. An immediate fall in the density of the juice notifies the workman of his error. The failure of the cell number to correspond with the number automatically registered notifies the chemist or superintendent of the error. To illustrate the above-mentioned error is appended a transcript of the battery report for two watches, December 9. The numbers in the column headed "Temperature" indicate the temperature of the juice at the time of determining its density, and not necessarily at the time of drawing the charge.

It will be noticed in this report that the density of the juice began to fall rapidly at 5.48 p.m.

LIX.

MAGNOLIA PLANTATION.									
<i>Date</i> ,—December 9.					<i>Watch</i> .—Second day and first night.				
<i>Battery-man</i>									
Cell No.	Time when Drawn.	Density.	Temperature.	Litres Drawn.	Cell No.	Time when Drawn.	Density.	Temperature.	Litres Drawn.
		° Baumé.	° C.				° Baumé.	° C.	
4*	1.21	6.0	38	1360	13	7.45	5.8	37	1360
5	2.06	6.1	37	1360	14	7.54	5.8	38	1360
6	2.15	4.6	50	1360	1	8.04	5.7	43	1360
7	2.25	5.3	38	1360	2	8.15	5.4	48	1360
8	3.34	5.2	37	1360	3	8.24	5.5	45	1360
9	3.49	4.2	50	1360	4	8.33	5.4	47	1360
10	4.12	5.0	40	1360	5	8.42	5.5	47	1360
11	4.22	5.0	39	1360	6	8.53	5.5	48	1360
12	5.21	4.3	50	1360	7	9.02	5.6	46	1360
13	5.30	4.9	55	1360	8	9.11	5.7	46	1360
14	5.39	3.4	57	1360	9	9.20	5.7	46	1360
1	5.48	3.3	50	1360	10	9.29	5.8	46	1360
2	5.57	2.0	58	1360	11	9.38	6.0	43	1360
3	6.06	1.8	57	1360	12	9.59	6.1	43	1360
4		Not drawn.			13	10.08	5.6	50	1360
5	6.25	3.9	44	1360	14	10.17	5.6	49	1360
6	6.35	4.2	40	1360	1	10.34	5.6	49	1360
7	6.45	4.5	41	1360	2	10.44	5.2	52	1360
8	6.58	5.0	37	1360	3	10.55	5.1	52	1360
9	7.07	5.2	36	1360	4	11.14	5.4	46	1360
10	7.16	5.5	36	1360	5	11.23	5.6	46	1360
11	7.25	5.6	38	1360	6	11.43	6.0	40	1360
12	7.35	5.8	37	1360	7	11.53	5.4	46	1360

* Work very irregular during second watch, 1.21 to 6 p.m., on account of trouble with the cane-cutter.

This sudden fall in density is due to more than one draw being made from one cell, or, in other words, the workman neglected to close a certain valve connecting with the juice main, and hence, instead of drawing from the cell last filled with fresh chips, drew repeatedly from a preceding cell through this neglected valve. The battery-man coming on duty the first night watch detected the errors from his predecessor's report, and corrected it. Considerable irregularity in the recorded density of the juice is due to great variations in the temperature at which the reading was made.

It is perhaps needless to add that the battery-man who made

these errors was relieved from duty the following day when he carelessly repeated the above mistakes. It would be easy for a workman to conceal his error by making a false entry in his report. The use of the automatic registering apparatus would effectually prevent or detect such false entries.

Delays due to encrustation on Yaryan.—The diffusion battery having been used three days continuously, it was decided to clear the yard and sugar house and begin test runs. These runs began December 1, and were continued until the end of the season. In this time there were few delays chargeable to the battery. There were numerous delays caused by the inefficiency of the cutters and the extremely foul condition of the Yaryan quadruple effect (p. 234). This latter failed to work up to its guaranteed capacity on account of a thick deposit of scale on the tubes. Late in the season Mr. Yaryan visited the plantation, and recommended boiling out the pans with caustic soda. This treatment was very effective, and the capacity was soon amply sufficient for the work required. In preparation for further enlarging his sugar house, Governor Warmoth has contracted for an 18-coil quadruple effect of the Yaryan system.

Some considerable delay was caused at the beginning of the season on account of the clarifiers not being in readiness. It was Governor Warmoth's intention to depend entirely on the clarification of the juice in the diffusers. This work was unsatisfactory, so he returned to the ordinary method.

Manipulation of the diffusion battery.—Fill two or three cells with water heated to near its boiling-point. Let these cells precede cell No. 1, *i.e.* the first cell filled with fresh chip. For convenience of reference the cells are referred to in numerical order, invariably calling the one in immediate connection with the water supply No. 1, and that containing fresh chips (after the first round of the battery) No. 12. No. 13 is open for the discharge of exhausted chips, and No. 14 is filling with fresh chips. By a proper manipulation of the valves force water into the first of the cells containing hot water, driving the water forward and into cell No. 1 at the bottom. By admitting the water at the bottom of the cell, the air is driven out at the vent in the cover. In the meantime cell No. 2 is filled with fresh chips. When No. 1 is full of juice the valves are changed, and the circuit established through the valve connecting with the upper part of the diffuser. The valve connecting with the bottom of No. 2 is then opened, and the juice from No. 1 passes in at the bottom of this cell, water taking the place of this juice. Cell No. 3 is filled with chips, and the same operations are repeated, and so on, until six or seven cells are filled. The number of cells so filled is dependent largely on the temperature of the water entering cell No. 1 and the probable extraction. Let us assume that seven cells have been filled. A charge of juice must now be drawn. The juice having passed

through seven cells of chips, no draw having been made, has about reached its maximum density. The work is now continued, a charge of juice being drawn from each cell filled. When cell No. 12 is reached, the hot water in No. 13 is discharged into the ditch; while No. 13 is filling, the water in No. 14 is discharged. The first round of the battery is now completed. The chips in No. 1 have been treated twelve times with fresh water and are now ready to be rejected. While cell No. 14 is filling with fresh chips the exhausted chips in No. 1 are being removed. This routine continues without variation. A few hours' practice at a battery is sufficient to train an intelligent labourer to do this work.

Influence of the dimensions and form of the cell.—If we place cuttings of cane in a vessel and surround them with water, no matter what may be the size or shape of the vessel, an equilibrium will soon be established, and the diluted juice bathing the chips will be sensibly of the same density as that contained in the cuttings themselves.

If, in the construction of a diffusion cell, we give it a diameter of 4 feet and a depth of but a few inches, there is no reason why the extraction should be either better or poorer than in a cell a few inches in diameter and several feet long, provided the circulation is equally good in each case. It is this proviso which should control the dimensions and form of a diffusion cell, and not the possibility of an increased or diminished extraction through variations in length of the column of chips which the water must traverse. The length of the column of chips has no influence whatever upon the extraction, but should not be sufficient to impede the circulation.

In the manufacture of sugar from beets there is a serious objection to a large cell, hence the tendency to make a capacity of 300 tons per day per battery a limit. This objection is the liability of the beet cuttings packing or matting, and thus interfering with the circulation. In the diffusion of cane even at high temperatures we find no such tendency to matting. The extraction in the Magnolia battery this season was very uniform, notwithstanding the increased length of the cells.

In cells of large diameter there is a possibility of difficulty in uniformly distributing the juice. The experience in cane work has been so limited, and so few batteries have been erected where a careful study of the work has been made, that we have little data on this point. A cell of small diameter can be built for less money than one of the same cubical contents but greater diameter. This refers especially to large batteries. The lower doors of a cell of large diameter should have more than two supports, namely the hinge and latch, in order to prevent springing.

If the length of the cell is excessive the great length of the column of cane chips will retard the current of juice, and it will be necessary to increase the water pressure.

The essential conditions that must be observed in the construction of a diffuser are that the form and dimensions must be such as to secure the best circulation of the juice through the chips.

Clarification by liming in the diffusion battery.—The first few days of the season 1888–89 at Magnolia, an attempt was made to use lime in the cells to neutralise the acids of the juice. The temperature of the three cells preceding that containing the fresh chips was maintained at as nearly 95° C. (203° F.) as possible. The results may be summed up briefly as follows. The diffusion juice was bright and perfectly clear. In order to be certain that the clarification was complete, the juice was run into the clarifiers and heated to the boiling-point. Quite a “blanket” formed, considering the preliminary clarification which had already been made. These impurities evidently resulted from the partial clarification that had taken place in No. 12 cell of the battery. The fresh chips being very much colder than the juice coming from the preceding cell, lower the temperature below the point to a good clarification. At the beginning of this work it was suggested to draw from the second cell from the last, *i.e.* No. 10. The juice from this cell, having been heated to the highest temperature practicable in the battery, is thoroughly clarified. This plan was not adopted, since it reduces the number of cells under pressure to 10, and necessitates driving two cells of juice ahead. The juice in these cells soon reaches its maximum density, and serves to heat the chips to such a temperature that a good clarification can be obtained.

An attempt was made to heat the chips in cell No. 12 in the following manner. Cell No. 12 was filled with chips and juice in the usual manner, except that compressed air was used to force the juice through the cells without changing the main battery valves, the air vent on No. 1 was opened, that on No. 12 closed, and the current reversed, forcing air into No. 12. The air vent on No. 12 was again opened, and the cell filled with juice, as usual in regular work.

It may be seen that the chips in No. 12 were twice bathed in hot juice, the first time raising their temperature considerably, and the second sufficiently high for clarification. The draw was made from No. 12 as usual.

This method of working, although it accomplishes the desired result, was too complicated, hence was not adopted.

Notes on the use of lime in the diffusion battery.—Fromentin advises “the use of a small quantity of lime in the diffusion of beets, 2 to 3 litres of milk of lime at 25° Baumé being added per diffuser. An increase in the purity of the juice and a better extraction are obtained.” Fromentiu cites an experiment in which the purity of the juice was increased 2 degrees.

In the above experiments a complete clarification of the juice was not claimed. Subsequent treatment by the carbonatation process was necessary.

In 1883 O. B. Jennings, of Honey Creek, Wis., was granted letters patent for certain processes in sugar manufacture, in which he specifically states that he uses either dry lime or lime whitewash mixed with the cane cuttings for the purpose of obtaining a thorough defecation in the diffusion apparatus. Mr. Jennings also claims that this process is applicable in the diffusion of sugar cane.

Lime was used in the diffusion cells at Wonopringo, Java, in 1887, for the purpose of clarification.

Lime was also used in the diffusers at the Planters' Experiment Station, Kenner, La. Dr. W. C. Stubb's experiments with this process, which were made entirely independent of the work of other experiments, were very successful.

This method of clarification is discussed in U.S.A. Bulletin No. 20, Division of Chemistry.

A few experiments were made at Magnolia, on a small scale, to determine how perfect a clarification can be obtained by this process. A pressure flask, such as is used in analytical work, was nearly filled with cane chips; sufficient lime was added to neutralise the acids in the juice, and the flask was finally filled with clarified diffusion juice, closed, and heated ten minutes to a temperature of 95° C. (203° F.). The flask was cooled, opened, and the juice was filtered through linen cloth. The filtered juice was then heated to its boiling-point in the open air. It remained perfectly clear, and even on boiling did not show signs of turbidity.

The conditions of this experiment were the same as those existing in regular diffusion work, except that in the latter case it is impossible to heat the last cell to as high a temperature as that obtained in the experiment. This experiment shows that, as soon as we obtain some simple method of carrying the juice in the last cells of the battery to a sufficiently high temperature, we can obtain a clarification superior to that obtained in the ordinary manner.

The use of lime is especially to be recommended when the work is irregular or the cane damaged by frost and subsequent fermentation.

WORKING TEMPERATURE OF THE BATTERY

Owing to the thickness of the chips furnished by the cutter it was necessary to work the battery at a high temperature, in order to obtain as good an extraction as possible with a low dilution. There is a decided inconvenience in working at high temperatures, due to the liability of the juice boiling in the heaters and steam collecting in the cells. It is possible for steam and air to collect to a sufficient extent to interfere with the circulation of the juice near the top of the cell. In the preliminary work with the diffusion battery the temperature of cells Nos. 8, 9, and 10 was maintained as nearly as possible at 85° C. (185° F.). The juice from cell No. 11 was kept as hot

WORKING TEMPERATURE OF THE BATTERY 315

as consistent with rapid work. The temperature of cells Nos. 2 to 7 inclusive ranged from about 65° to 70° C. (149° to 158° F.). With thin chips and a moderate dilution the extraction was very good. Owing to a lack of cutting capacity it was necessary to increase the thickness of the chips and work the battery at a very much higher temperature. In the early part of the work the water for the battery was passed through a large heater; later on, it was found that as good results could be obtained without the heater as when using it.

The range of temperature during the greater portion of the season is shown in the following table. It must be remembered that the battery had fourteen cells, twelve of which were in activity, one filling and one emptying. The small amount of heating surface in the calorisor should also be taken into account.

LX.—TABLE SHOWING COMPARATIVE TEMPERATURE IN EACH CELL IN SUGAR-CANE AND SUGAR-BEET DIFFUSION BATTERIES.

Cell Number.	Sugar Cane.		Sugar Beet.	
	Temperature.		Temperature.	
	° Cent.	° Fahr.	° Cent.	° Fahr.
1	60	140	40	104
2			60	140
3	}	70-80	}	176-185
4				
5				
6				
7				
8	95	203	}	80-85
9	95	203		
10	95	203		
11	85	185	65-70	149-158
12	70	158	40-50	104-122

The low temperature of cells Nos. 1 and 2, 11 and 12, in the second table is noticeable. In cells 11 and 12 this low temperature is due to the moderately heated juice in cells 3 to 10 coming in contact with the cold beet cuttings. The temperature of cells 3 to 10 is purposely maintained at a moderate degree, to prevent the cuttings from swelling and impeding the circulation. Even if it were practicable, so far as the capacity of the calorisors is concerned, to raise the temperature of cell No. 1 above 40° C. (104° F.), it would be decidedly objectionable on account of the effect of high temperatures on the beet cuttings. Practice has demonstrated that a temperature above 40° C. in this cell is liable to seriously interfere with the pressing of the exhausted cuttings.

Is it not possible that the high temperature at which we usually conduct cane diffusion is largely the cause of the difficulty that has been experienced in milling the exhausted chips (see p. 301)? In the experiments made in 1887 at Magnolia considerable difficulty was experienced in milling the chips; so much, in fact, that the experiment was practically a failure. In the battery work the water entering cell No. 1 was heated to about 71° C. (160° F.), and by the time it reached the second cell its temperature was little below the boiling-point. On the contrary, this season, when the milling experiment was very successful, the temperature of the first cell did not exceed 60° C. (140° F.).

Owing to the trouble with the cutter, we had no further opportunity to make experiments in conducting the battery work at different temperatures. With thin chips, however, the maximum temperature need not exceed 85° C. (185° F.). There is little doubt but that the relatively lower purity of the diffusion juice as compared with the normal juice was due to the high temperature at which it was necessary to work.

Juice extracted at a temperature of 85° C. (185° F.) admits of easier and more thorough treatment in the sugar house.

Dilution.—Two methods of stating the dilution of the normal juice are employed in this report, namely *apparent dilution* and the *actual dilution*. In addition, the extra evaporation in terms of the diffusion juice is also given.

Owing to the frequent variations in the juice content of the cane we have a variable dilution even with a constant draw. For the same reason we have a variable reduction in the percentage of sucrose in the juice, aside from irregularities of extraction. The relation of the diffusion drawn to the actual amount contained in the cane is the apparent dilution.

It has been customary in diffusion work in U.S.A. to arbitrarily assume a juice content of 90 per cent. of the cane, and reduce this weight to volumetric terms based upon the density of the normal juice; a comparison of this volume of juice is then made with the volume of diffusion juice drawn. The actual volume of juice in the cane is compared with the volume of diffusion juice drawn, and the result is termed the *apparent dilution*. The nearer we approach a perfect extraction, the nearer the apparent dilution approaches the actual.

The *actual dilution* is the proportion of water added to the normal juice to reduce its percentage of sugar to that of the diffusion juice, hence the actual dilution represents the evaporation necessary per cent. normal juice to remove the added water. In calculating the dilution the sum of the percentages of sucrose and glucose (Table LXI.) are taken, in order to diminish the errors resulting from inversion.

DILUTION OF CANE DIFFUSION JUICE 317

RÉSUMÉ SHOWING THE AVERAGE OF MANUFACTURING RESULTS AT MAGNOLIA, 1888-89.

Cane, per cell (diffuser)	pounds	2760
„ per cubic foot of cell capacity	„	25·8
Diffusion juice drawn, per cell	gallons	339·2
„ „ per 100 pounds cane	pounds	107·8
„ „ „ „	gallons	12·3
Sucrose left in exhausted chips „	pounds per ton of cane	11·28
Actual dilution	per cent. normal juice	23·4
Apparent dilution	„ „	23·3
Additional evaporation	„ „ diffusion juice	22·1
„ „	pounds water per ton of cane	476·4
Extraction „	per cent. sucrose in the cane	94·72

LXI.—RÉSUMÉ SHOWING THE MEAN COMPOSITION OF THE NORMAL AND DIFFUSION JUICES, THE MAXIMUM AND MINIMUM DENSITY, PERCENTAGES OF SUCROSE, REDUCING SUGARS (GLUCOSE, ETC.), COEFFICIENT OF PURITY, ETC., 75 ANALYSES (MAGNOLIA, 1888-89).

	Normal Juice.			Diffusion Juice.		
	Means.	Maxima.	Minima.	Means.	Maxima.	Minima.
Degrees Brix	16·4	17·8	12·9	12·9	14·6	11·5
„ Baumé	9·1	9·9	7·2	7·2	8·1	6·4
Specific gravity	1·0672	1·0735	1·0523	1·0522	1·0596	1·0464
Sucrose per cent.	14·1	15·3	10·1	11·0	12·5	8·8
Reducing sugars (glucose, etc.) „	0·56	1·0	0·33	0·48	0·72	0·26
Coefficient of purity	85·97	89·50	78·27	85·27	91·88	76·47
Glucose per 100 sucrose	3·97	7·75	2·29	4·36	7·35	2·5
Inversion per cent. diffusion juice	0·055	2·96	...

Increase in the evaporation in the sugar-cane diffusion juice process compared with milling.—In figuring coal consumption in the comparison of mill and diffusion work, all statements should be based on the actual dilution. In comparing the figures on coal consumption of the Magnolia Diffusion Work (pp. 302-304), Mr. Spencer remarks that manufacturers should not neglect to note the exceptional richness of the juice, and further, that in the Magnolia plantation a quadruple-effect Yaryan system was employed. The following table shows the relative quantity of water to be evaporated in mill and diffusion work. The data show an approximate average of the season's work.

The estimation of the mill year is arbitrary, and is based on the previous work and the total juice in the cane.

LXII.—EVAPORATION IN DIFFUSION WORK AS COMPARED WITH THAT OF 72 PER CENT. MILLING.

Mill Work Total Evaporation.	Diffusion Work.			
	Evaporation due to the Dilution.	Evaporation due to Increased Extraction.	Total Evaporation.	Increase over 72 Per Cent. Milling.
Lbs. Water Per Ton. Cane. 1195·7	Lbs. Water Per Ton. Cane. 481·7	Lbs. Water Per Ton. Cane. 193·1	Lbs. Water Per Ton. Cane. 1870·5	Per Cent. 56·4

The above shows an actual increase in the coal consumption for evaporation of over 56 per cent., assuming that the entire fuel supply is obtained from coal. In addition to this increase there are still others, due to a larger product, to centrifugal, and to the greater surface exposed for radiation. The increased evaporation in quadruple should not require with good boilers well fired more than 175 lbs. additional coal per 1000 lbs. sugar.

Fuel consumption in cane and beet diffusion.—Comparing the results with that of the best equipped beet-sugar houses employing quadruple effects, Mr. Spencer says the latter, according to Horsin-Deon, burn 4·23 kilogrammes of coal per hectolitre of juice; comparing this with the Magnolia work, basing the figures on the same dilution, we have 100 lbs. of coal consumed per average of 417 lbs. in the cane-sugar house. Even the Austrian houses in their best work, where nearly twice as much diffusion juice is drawn per 100 lbs. of beets as was drawn from the cane at Magnolia, burn only 180 lbs. per 2000 lbs. of beets. These houses employ quadruple-effect evaporation with all Rillieux's improvements. The large fuel consumption at Magnolia cannot be charged to the Yaryan apparatus or to the vacuum pan. Repeated tests have demonstrated the high efficiency of the Yaryan. The vacuum strike pan is of the low-pressure type, and of the most modern construction. One cause of the excessive fuel consumption was, no doubt, the use of coal under boilers designed for an entirely different class of fuel. The fuel burned under the coal boilers as estimated by several experts was approximately 1 lb. per 6 lbs. of water evaporated. There was probably a large increase due to the wastage of the waters of condensation from the battery heaters. The beet houses cited do not employ bone black, hence the fuel consumed in preparing the liquors for filtration, etc., should be deducted in this comparison.

In the beet-sugar houses all the evaporation and heating of juices and syrups, etc., is in multiple effect. This is accomplished by the

FUEL CONSUMPTION IN CANE DIFFUSION 319

improved methods of Rillieux (p. 224 *et seq.*). All such work at Magnolia was in single effect except the evaporation.

All the available data in diffusion work indicate that with the very best modern appliances the fuel consumption need not exceed 100 lbs. of coal per ton of cane, or 500 lbs. per 1000 lbs. of sugar. This is as low a coal consumption as is obtained in the best American cane house economising the wet bagasse directly in their furnaces; but if the Germans and Austrians can work with this high degree of economy, cannot the Americans do so as well? The day is not distant when coal will be only required as an auxiliary in firing after stoppages, the exhausted chips furnishing the fuel required.

Planters estimating on diffusion and intending to use the evaporating appliances already in place for milling must not neglect to note that they will be compelled to work less cane per day, to compensate for the increased evaporation and extraction. In other words, they must enlarge the capacity of their houses in proportion to the increased yield and dilution.

In case the chips are not burned, at least two and one-half times as much coal must be provided for diffusion as would be for milling where in the latter case the bagasse is employed as fuel.

LXIII.—MAGNOLIA PLANTATION CROP REPORT, 1888, DIFFUSION RUNS.*

[Third run, December 1 to 8 inclusive: Cane worked, 1079·5 tons ; diffusion work.]		
Description.	Yield.	Sugar per Ton of Cane.
	lbs.	lbs.
First sugar (white)	120,085	111·24
Second sugar (yellow C)	56,805	52·62
Third sugar (waggon)	31,705	29·37
Fourth sugar † (waggon)	21,590	20·00
Total	230,185	213·23
[Fourth run, December 9 to 22 inclusive: Cane worked, 1799 tons ; diffusion work.]		
First sugar (white)	152,881	84·98
Second sugar (yellow C)	160,002	88·94
Second sugar (waggon)	12,037	6·69
Third sugar (waggon)	71,059	39·50
Fourth sugar † (waggon)	35,980	20·00
Total	431,959	240·11

* Cp. Table LVI. p. 288.

† See note marked * on following page.

LXIII.—MAGNOLIA PLANTATION CROP REPORT, 1888, DIFFUSION RUNS—
continued.

[Fifth run, December 23 to January 14, 1889 : Cane worked, 3062 tons ; diffusion work.]		
Description.	Yield.	Sugar per Ton of Cane.
First sugar (white)	234,135	76·46
Second sugar (yellow C)	246,464	80·49
Third sugar (waggon)	114,825	37·50
Fourth sugar* (waggon)	61,240	20·00
Total	656,664	214·45

* One-half of this sugar was estimated from the half swung out. Fourths are divided between milling and diffusion in proportion to the yield of the other sugars.

Crop, total	pounds	1,855,230
Crop, average	pounds per ton of cane	203·1
Diffusion, average	" "	222·0
Mill, average	" "	167·9
Diffusion, increase	" "	54·1

The method of sampling pursued in former seasons was the only one available. 100 c.c. of juice were taken from each charge and stored in a large bottle until a sufficient quantity for a fair sample had been taken. A handful of fresh and one of exhausted chips were also taken from each cell. The former was passed through a small hand mill, and the juice so obtained was analysed, and the analysis taken to represent the normal juice of the cane.

The run numbers were continued from those of the mill work which preceded diffusion. During the third run (that is, the first diffusion run) numerous experiments were made to determine the best method of conducting the battery work. The thickness of the chips soon necessitated work at high temperatures, and compelled the abandonment of further experiments.

The yield of sugar in proportion to the sucrose present was larger in subsequent runs. This is partly due, in addition to richer cane, and in the fourth run a better extraction, to losses resulting from the fermentation of the syrups. This fermentation was due to two causes. The failure of the vacuum pan traps to operate satisfactorily prolonged the boiling from 10 to 12 hours, 5 hours usually being sufficient time in which to boil a strike. This caused an accumulation of syrup in the storage tanks. At this time a supply of new bone black was substituted for the chars, which had been in use for many years. The sugar-maker never having had experience in the use of new bone black, was unprepared for the difficulties in

its use. For criticism of this method of working, see p. 356. The facilities for washing bone black at Magnolia are very crude, hence the char was sent to the filters badly washed. On the admission of the hot syrups to the filters dense volumes of ammonia filled the room. The syrups so filtered fermented with extreme rapidity, entire tauks of syrup showing a viscous fermentation in two or three hours. It was often impossible to centrifugal syrups grained in the pan from these syrups, hence several strikes were boiled to string-proof and sent to the hot room. This fermentation and the consequent difficulties in manufacture undoubtedly caused a large loss of sugar. The following comparison of the yield for the three diffusion runs indicates the extent of this loss:—

LXIV.—AVAILABLE SUCROSE (SUCROSE MINUS $1\frac{1}{2}$ TIMES GLUCOSE).

Run Numbers.	Available Sucrose. Lbs. per Ton of Juice.	Yield of Sucrose. Lbs. per Ton of Juice.	Available Sucrose obtained in Sugars per Cent.
Third .	222.7	203.63	85.66
Fourth . . .	243.3	228.73	94.01
Fifth	230.8	204.39	80.56

The degree of extraction was practically the same in the third and fifth runs, the cane was considerably better in the third, the glucose per cent. being respectively 3.40 and 4.17. The necessity of apportioning the fourth sugars equally among the diffusion runs probably gives the third run a better record than it should have. The difficulty in estimating this loss emphasises the necessity of facilities for keeping the products from each run separate, if one desires a thorough chemical control.

Owing to the molasses from the entire season's work being mixed in the cisterns, and several losses known to have taken place, but which could not be determined, the inversion could not be calculated for this or the other runs. The lower proportionate yield in the fifth run was probably due to methods of manufacture.

LXV.—RELATION BETWEEN THE DENSITY OF THE JUICE OF THE SUGAR CANE AND ITS RICHNESS IN SUGAR.

Degrees Baumé at 15° C.	Weight of 1 Litre at 15° C. in Grammes. Density Water as 1000.	Sugar per Decilitre Juice. Lbs. per 10 Imp. Gals.	Sugar per Decilitre of pure Sugar Solution. Lbs. per 10 Imp. Gals.	Purity of the Juice.	Coefficient to obtain the Sugar per Decilitre of Juice.*	Sugar per Cent. of Juice.	Sugar per Cent. of Cane.
7·25	1053	10·3	14·02	73	1·94	9·8	8·4
7·35	1054	10·6	14·28	74	1·96	10·0	8·6
7·50	1055	10·9	14·54	75	1·98	10·3	8·9
7·65	1056	11·2	14·80	76	2·00	10·6	9·1
7·80	1057	11·55	15·06	76	2·03	10·9	9·4
7·90	1058	11·9	15·32	78	2·05	11·2	9·7
8·00	1059	12·2	15·58	78	2·07	11·5	9·9
8·15	1060	12·5	15·84	79	2·08	11·8	10·1
8·30	1061	12·8	16·10	79	2·10	12·1	10·4
8·40	1062	13·1	16·36	80	2·12	12·3	10·6
8·50	1063	13·4	16·62	81	2·13	12·6	10·8
8·65	1064	13·7	16·88	81	2·14	12·9	11·1
8·80	1065	14·08	17·14	82	2·16	13·2	11·4
8·90	1066	14·4	17·40	83	2·18	13·5	11·6
9·00	1067	14·7	17·66	83	2·19	13·8	11·8
9·15	1068	15·0	17·92	84	2·20	14·0	12·1
9·25	1069	15·3	18·18	84	2·21	14·3	12·3
9·40	1070	15·6	18·45	85	2·23	14·6	12·5
9·55	1071	15·9	18·71	85	2·24	14·8	12·8
9·65	1072	16·2	18·97	85	2·25	15·1	13·0
9·80	1073	16·55	19·23	86	2·27	15·4	13·3
9·90	1074	16·9	19·49	87	2·28	15·7	13·5
10·05	1075	17·2	19·75	87	2·29	16·0	13·8
10·15	1076	17·5	20·01	87	2·30	16·3	14·0
10·25	1077	17·8	20·27	88	2·31	16·5	14·2
10·40	1078	18·1	20·53	88	2·32	16·8	14·4
10·55	1079	18·4	20·79	88	2·33	17·1	14·7
10·65	1080	18·75	21·06	89	2·34	17·4	14·9
10·80	1081	19·1	21·32	89	2·35	17·7	15·2
10·90	1082	19·4	21·58	90	2·36	17·9	15·4
11·05	1083	19·7	21·84	90	2·37	18·2	15·6
11·15	1084	20·0	22·10	90	2·38	18·45	15·9
11·25	1085	20·3	22·36	91	2·39	18·7	16·1
11·40	1086	20·6	22·62	91	2·40	19·0	16·3
11·55	1087	20·9	22·88	91	2·40	19·2	16·5
11·65	1088	21·25	23·14	92	2·41	19·5	16·8
11·75	1089	21·6	23·40	92	2·42	19·8	17·0
11·90	1090	21·9	23·67	93	2·43	20·1	17·3
12·05	1091	22·2	23·93	93	2·44	20·3	17·5
12·15	1092	22·5	24·19	93	2·45	20·6	17·7
12·25	1093	22·8	24·45	93	2·45	20·9	17·9
12·35	1094	23·1	24·71	94	2·46	21·1	18·2
12·50	1095	23·45	24·98	94	2·47	21·4	18·5

* See explanation of Table XVII. on p. 51: *e.g.* 1·053 sp. gr. = 5·3 Excise degrees, and 5·3 × 1·94 = 10·3 of sugar.

CHAPTER XI

DEFECATION OF CANE JUICE—THE EVAPORATION OF THE CANE JUICE TO THE CRYSTALLISATION-POINT

THE CANE JUICE AND ITS PROPERTIES

THE cane juice as it comes from the mills is a straw-yellow opaque liquid, often also of a dull grey or olive-green colour, which decomposes very rapidly, assuming a blackish tint. Its taste is balmy and sweet. It exhales the balsamic odour of the cane. The density varies from 1.033 to 1.106, according to circumstances. The juice, in fact, varies with the nature of the soil, with the season, the variety of the plant, and the manuring, whether extensive or the reverse. The fresh and more or less turbid juice may be separated into two portions—a liquid and a solid. The suspended solid portion may be separated from the liquid in which it is suspended by filtration or settling. The solid ingredients of the pressed juice consist of the cellular parenchyma of the cane, its woody fibres and bark, mixed with a green colouring principle similar to, if not identical with, chlorophyll. 100 parts of cane yield 98 to 110 parts of juice, according to the skill of the manufacturer and the quality of the cane.

The juice requires speedy clarifying treatment on account of its liability to the acetous fermentation.—In equatorial climates the expressed juice when left to itself is spontaneously liable to the acetous fermentation. Even such a short period of time as twenty minutes often suffices to induce this destructive fermentative action. To obviate this it is necessary to immediately proceed to clarify it.

The clarified liquid still subject to the alcoholic fermentation.—Even when the liquid has been clarified by the removal of its green fecula and albuminous matter, it is still liable to ferment. But the nature of the fermentation is now different; instead of an acetic acid fermentation, the liquor is now liable to an alcoholic fermentation. Only such clarifying processes, therefore, as act in the desired manner as promptly as possible should be adopted.

This fermentation formerly mistaken for a natural purification process.—Now the West Indian sugar factory managers, seeing this blackish liquid rise rapidly to the surface in the form of a scum, carrying with it all the impurities of the juice to the top of the

liquid, were accustomed to let this scum form, believing that it constituted a natural purification of the juice, whilst all the time it was fermentation that had started. And this fermentation was accompanied by a considerable formation of glucose at the expense of the sugar. This custom was therefore a deplorable one. Moreover, in cane-sugar factories reforms are necessary at every step. This is due to the complete ignorance of the manufacturers. It is not long since chemists were introduced into these torrid countries where mediocrity reigns supreme over the complete industrial vacuity of the natives, and it is only after extraordinary exertions that they have been able to obtain a footing by fighting against the routine of the sugar-foremen.

Improved results from rational working.—These efforts have already produced remarkable results. On those plantations where they have been located the yields have considerably increased, but in spite of that example, which must be seen by the most wilfully blind, very few planters follow suit.

Neutralising and boiling the juice.—The juice as it comes from the mill should therefore be treated forthwith. In fact, all ferments are destroyed at 100° C. (212° F.). Thus all destruction of sugar by ferments is avoided.

The effect of the natural acidity on the sugar during boiling.—But cane juice is naturally acid, and if an acid saccharine liquid be heated the sugar is *inverted*. It is therefore necessary to both boil and neutralise it at one and the same time. This process is what is termed *defecation*, this boiling having further functions still.

Boiling and liming.—The juice to be defecated is run into a jacketed pan, called the *defecation* pan. Steam is immediately run into the jacket. The liquid heats, and when it reaches about 80° C. (176° F.) 0.002 to 0.003 per cent. of lime, in the condition of milk of lime, is added and heat continued to be applied.

The albuminous and waxy principles present in the juice.—The cane juice contains albumen in abundance, and also other impurities which come from the pressure in the mill of the bark mixed with the vesicular tissue, such as the cerosin, a sort of waxy substance which it is necessary to separate. A description of cerosin is given on p. 284, and illustration on p. 283, Fig. 63, A.

The liquid being saturated by lime and brought to the boil, a scum is soon seen to form on the surface. It is the albumen which is coagulated, and which brings in its train (1) the excess of lime, (2) the cerosin, (3) the suspended matter, (4) in fact, all the substances which it is necessary to eliminate. There is thus formed an abundant scum, which is removed by skimmers. But heat must not be applied for too long a time, because the pan would prime and overflow completely. A gutter is even arranged all around the pan to collect any froth or foam which may prime over at that time.

Fractional separation of the defecated liquor.—When the steam is turned off the whole is allowed to settle. If the liquid be sampled at this moment at different depths it will be found that the centre is quite limpid, whilst the bottom and the surface are charged with suspended matter. Accordingly, by means of a tap with holes at different heights, the whole of the clear liquid is allowed to flow away, then the bottom and the upper portions are run into tanks to be passed to the filter presses.

The clear liquid, as well as that which flows from the filter presses, are now fit and ready for evaporation. It will be seen how simple it is to treat the cane juice. It evolves no carbonatation or excess of lime. *Simple boiling after neutralisation by lime is all that is usually required in the purification of cane juice.*

The juice from the sugar-cane diffusers still more easily treated.—The sugar-cane diffusion juice is much easier still to work, because it is almost fit to evaporate just as it comes from the diffusers. Therefore they dispense with defecation in some instances, if the juice has been treated in the diffusers themselves by a defecating agent. In any case, a slight ebullition with a trace of lime suffices.

The former dark-coloured juices entailed the use of bulky bone-black filters.—When the cane juices were so badly wrought that they were full of glucose and colouring principles, energetic filtration through animal charcoal was necessary in order to get them to crystallise. At that time the sugar factories contained numerous charcoal filters, and the planters took enormous pride in showing the great extent of the filtration equipment of their factories.

Bone-black filters at the present day a sure sign of bad working.—At the present time it is all the other way about. A sugar factory containing a great number of bone-black filters is a badly conducted one, because modern equipped sugar factories do not now use bone-black filters, which are now confined to the refinery.

The suspended matters the chief impurity to be eliminated.—Everywhere mechanical filters are to be seen; the juice as it comes from the mill or the diffusion—that coming from the defecation pans or the filter presses—are all filtered through bagasse or cloth to free them from suspended matters, the real enemies of good working.

CAMBRAY'S PROCESS FOR THE RATIONAL PURIFICATION OF SUGAR-CANE JUICE.

Although from a mechanical point of view the great progress made in the sugar-beet industry has extended itself to the manufacture of sugar from the sugar cane, yet this is not the case in regard to the yield of sugar obtained, owing to the want of a rational process for purifying the cane juice. The latter contains, besides sugar, variable quantities of glucose, gummy substances, etc., which exert

a great retarding influence on the crystallisation of the sugar and induce the rapid fermentation of the latter. That is why the amount of glucose increases throughout the different stages of manufacture at the expense of the crystallisable sugar. The object of Cambray's process, therefore, is to eliminate the gums and the principal mineral and organic impurities, thus reducing the glucose present and avoiding any future formation thereof. The yield is increased and the final quantity of molasses reduced to a minimum.

The process consists in the double sulphitation of the cane juice in the presence of lime; from $3\frac{1}{2}$ to 4 lbs. of quicklime, in the form of milk of lime, is added to every 100 gallons of the juice as it comes from the mills or the diffusers, the temperature of the juice being kept between 50° to 55° C. (122° to 131° F.). A current of gaseous and anhydrous sulphurous acid, produced by means of a special apparatus (Fig. 74), is then passed through the liquid, and the sulphitation continued till the filtrate gives an alkalinity corresponding to 0.2 to 0.3 gramme of lime per litre; the turbid juice is then run into decanting tanks. The muddy matters settle quickly, and the juice may be readily decanted. The sludge is sent to the filter presses, and the clear juice is brought to the boil and again treated with sulphurous acid until it is slightly acid. The sulphited juice is not only much purer than the crude juice, containing less glucose, etc., but it is much less liable to fermentation than the latter, thus facilitating subsequent operations. It is very easily evaporated and boiled down, and four successive crystallisations may be obtained from it, the sugar being of high quality. The following results were obtained in a cane-sugar factory which used this double sulphitation process in 1895, the ordinary processes having been previously followed:—

		1894.	1895.	
Purity	. . . {	Diffusion juice purity	77.86	82.96
		Syrup purity	76.60	84.62
		Difference	- 1.26	+ 1.66
Non-saccharine	. . . {	Non-saccharine, diffusion juice	27.79	20.52
		Non-saccharine, syrup	30.04	17.95
		Difference	+ 2.25	- 2.57
Glucose per cent. of sugar	. . . {	Diffusion juice	13.48	7.17
		Syrup	14.53	4.89
		Difference	+ 1.05	- 2.28

These figures speak for themselves. Identical results have been observed in the composition of the products of later stages:—

		1894.	1895.	Difference.
Seconds—Purity	48.19	71.21	+ 23.02
„	Glucose per cent. of sugar	51.80	12.51	- 37.29
„	Non-saccharine per cent. of sugar	103.26	40.54	- 62.72
Thirds—Purity	35.27	56.18	+ 20.91
„	Glucose per cent. of sugar	91.93	25.87	- 66.06
„	Non-saccharine per cent. of sugar	183.30	78.27	- 105.03

The average analyses of the *masses cuites* of the first and second sugars are as follows:—

	1st <i>Masse</i> <i>Cuite.</i>	2nd <i>Masse</i> <i>Cuite.</i>
Degrees Baumé	48·16	47·6
„ Brix	92·7	91·2
Sugar	78·92	61·8
Water	10·05	13·53
Glucose	3·99	8·5
Ash	3·28	7·25
Organic matter	3·73	6·92
Purity	87·77	73·16
Glucose per cent. of sugar	5·05	13·75
Saline coefficient	24·06	8·52

SULPHITATION PAN

This pan (Fig. 75) is made of very strong wrought iron, with the bottom sloping towards the discharge valve so as to ensure as complete evacuation as possible. It is furnished with a steam coil, a

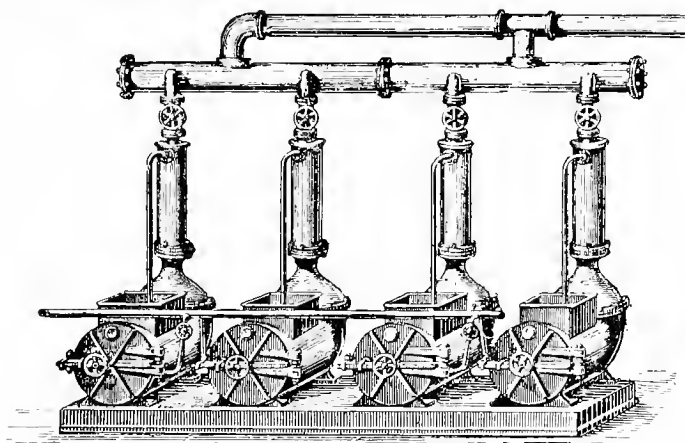


FIG. 74.—Sulphur kiln battery (CAMBRAY).

steam valve, a juice tap, a gauge tap, and a discharge tap, wrought by a wheel attached to a rod, which is actuated on from the upper part of the pan. These pans may be made to work a layer of liquid a metre in depth or more.

SULPHITATION OF THE SYRUP

Much sulphurous acid is used in cane-sugar factories as a decoloriser and defecating agent for the syrups. This sulphitation produces very good effects both on the colour and the yield of sugar.

EVAPORATION

The old method of boiling down the juice in a row of open pans set over a furnace and its flue.—The defecated juices have to be evaporated and boiled down to the crystallising point. Although there is no necessity to dwell on the subject, we must here say a few words about the ancient naked-fire methods which still subsist in the old factories. The process has been described in the older manuals until the descriptions are about as stale as the process itself is wasteful. The juices are run into a series of pans placed in single file on

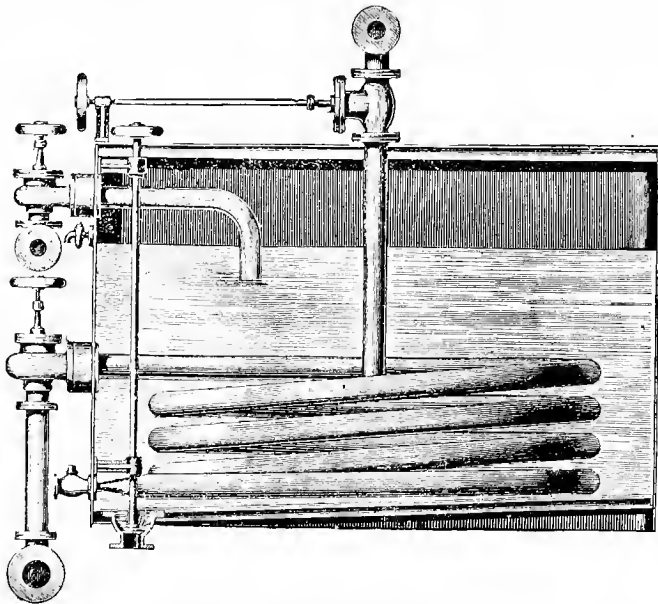


FIG. 75.—Sulphitation pan (CAMBRAY).

the same furnace. The first, the largest pan, which on that account is called the *grand*, is immediately above the fire. The others, which gradually become smaller, are to the rear; and the last of all, the smallest, is next to the chimney, where the flame is weakest.

The juice is run into No. I., No. II. is charged from No. I., No. III. from No. II., No. IV. from No. III. The juice thus becomes denser and denser until it reaches the last, when it is in the condition of syrup.

Claying.—Formerly this syrup, when well concentrated, was poured into sugar-loaf moulds, then carried into the crystallisation

room, where it solidified on cooling. This mass was then *clayed*, that is to say, its surface was covered by moistened clay. This moisture percolated through the loaf to its apex, driving the molasses before it.

When the mould was undone, the paste, covered with clay, was removed, the apex full of molasses was detached, and a more or less yellow loaf was left, which represented the purest product of the manufacture.

Concentrating the juice in open-air pans heated by steam.—Later on, evaporation was effected by steam in the open air. The most improved appliances were Wetzell's pan and Chenailier's pan. In the middle of a trough filled with cane juice a hollow axis revolved, furnished throughout its entire length by hollowed-out enlargements in the form of lenses. Steam circulated in the axis, and filled the lenses, which came half out of the liquid.

The juice moistening the lenses, being unceasingly renewed by the rotation and by some receptacles fixed on their exterior which were discharged from above, was in the form of this thin layer evaporated very quickly, and soon was converted into syrup fit for boiling. Boiling was then done in Howard's vacuum pans, the first which were wrought in a vacuum.

At the present time evaporation is effected by means of the triple and quadruple effect, as in beet-sugar works, and the remainder of the work in no way differs from that already described: boiling, turbinng, etc., as in the case of beets.

MOLASSES

The last cane-sugar molasses are sold to the distilleries, for the manufacture of rum or for general consumption. Cane-sugar molasses have, in fact, a perfumed taste which is very agreeable, the opposite being the case with beet molasses. The natives of sugar-cane countries, and even the British and sailors in general of all countries, consume enormous quantities of it.

The second quality of cane sugar is yellow and has a delicious taste. It is accordingly consumed directly in this condition, and many prefer it to the first quality white sugar, in which this perfume is much less pronounced. But genuine "Demerara" is rare, whilst dyed imitations, often from poisonous dyes, abound.

LXVI.—TABLE SHOWING THE MEAN COMPOSITION OF FOURTH MASSES CUITES AND MOLASSES THEREFROM AT MAGNOLIA, 1888-89.

	Sucrose direct polari- sation.	Sucrose double polari- sation.	Glucose.	Coefficient of purity (direct polarisation).	Coefficient of purity (double polarisation).
Molasses.	Per Cent. 30·00	Per Cent. 33·93	Per Cent. 17·6	37·93	42·68
<i>Masses Cuites</i>	44·45	48·79	17·45		..

Such are the points to be noted in the manufacture of sugar from the cane, only the first stages of which differ from that of the beet. All the other processes are identical; the analyses are the same.

All the sugar that is not consumed on the spot is bought by the refiners. The cane-sugar market is therefore the same as the beet-

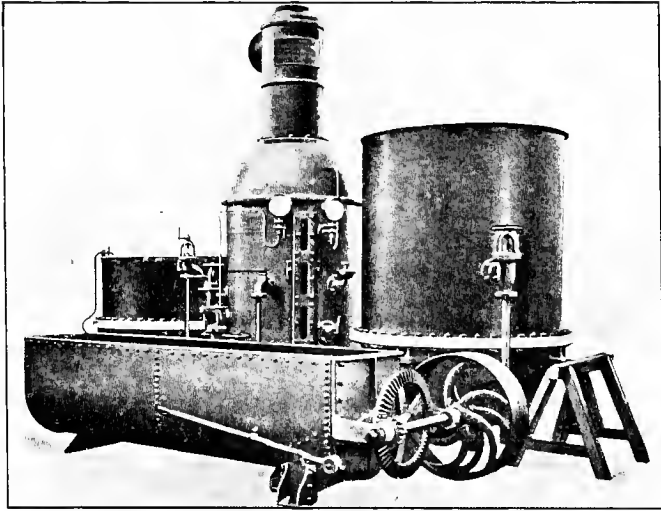


FIG. 76.—Compact cane-sugar plant for colonial homestead (C.A.I.L.).

sugar market. They thus follow the same quotations and the same fluctuations of the market.

The chemistry of molasses is dealt with on p. 382 *et seq.*

Fig. 76 shows a compact sugar-cane plant, suitable for the colonies, consisting of vacuum pan, defecation pan, cooler, etc.

CHAPTER XII

SUGAR REFINING

The raw material.—Most refiners, when at all practicable, buy their sugars in one out of three possible conditions only—

1. White sugar.
2. Yellow sugar, with 88 per cent. yield.
3. Brown impure sugar.

Some refineries buy sugar under one of these conditions only,—*e.g.* Russian refiners only treat white sugars, whilst certain Hamburg refineries only treat the black impure sugar from the Réunion. Again, the Clyde refiners, in their day, only bought sugar with less than 4 or 5 per cent. of fruit sugar.

The purchase and selection of the raw sugar.—It will thus be seen that several points require attention in selecting the raw material. The quality selected will, to a great extent, depend upon the method of refining in vogue in the refinery in which the raw sugar is to be treated. There are many varieties and qualities of raw sugar, each of which lends itself more or less to some particular method of refining. Moreover, it must be borne in mind that the bright yellow sorts are almost exclusively monopolised by the grocers for sale in the original muscovado condition. Hence the darker and duller tinted varieties are only available for the refiner's purpose. In some systems of refining, such as those in vogue in Greenock, when the sugar-refining industry attained its zenith in that town, no syrup was made. In such a case, therefore, it was absolutely essential to use sugars which contained only a comparatively small amount of uncrystallisable sugar—say, not exceeding, on an average, more than 3 or 4 per cent. On the other hand, where a considerable quantity of syrup is turned out, the quality of the sugar is not so important a matter, and low-quality sugars, such as *Syrup Mauritius*, *Jaggery*, and *Manilla*, may be used (Wallace). Again, judicious mixtures may often be made, such as beetroot sugar with sorts rich in fruit sugar; but where no syrup is made, as in the Greenock system, too large a proportion of beet must be guarded against, as the soluble saline matter has a tendency to become concentrated in the lower qualities of the refined article, not only hindering its crystallisation, but imparting to it a peculiarly nasty taste.

LXVII.—ANALYSES OF RAW SUGAR.

	Cuba Muscovado.	Grenada.	Trinidad.	Demerara.	St. Vincent.	Concrete.	Melado.	Molasses.	Pernambuco.	Paraiba.
Cane sugar .	92·35	92·31	90·41	90·80	89·00	84·20	67·00	47·0	88·31	84·90
Fruit sugar .	3·38	4·06	3·84	4·11	5·85	8·45	11·36	20·4	4·82	6·00
Extractive, etc.	·66	·66	·95	·77	·76	1·70	1·93	2·7	·94	1·28
Soluble salts .	·62	·37	·86	·92	·62	1·10	·76	2·6	·80	1·20
Insoluble . . .	·15	·04	·22	·20	·05	..	·15	..	·73	1·10
Water	2·84	2·56	3·72	3·20	3·72	4·55	18·80	27·3	4·40	5·52
	100·00	100·00	100·00	100·00	100·00	100·00	100·00	100·0	100·00	100·00
Colour, D. S. Cane sugar obtainable .	13	13	10	10	10	12	8	8
	85·9	86·4	82·3	82·1	80	70·2	51·8	13·6	79·5	72·9

	Bahia.	Syrup Mauritius.	Calcutta Date.	Jaggery.	Unclayed Manilla.	Amoy.	Formosa.	Maple.	Fr. Beet (fine).	Ger. Beet (low).
Cane sugar .	86·80	86·00	87·06	86·73	79·00	74·50	76·53	72·60	94·30	87·80
Fruit sugar .	5·03	6·35	6·95	6·05	11·76	16·13	13·38	13·95	·25	·33
Extractive, etc.	1·72	1·62	·65	1·29	1·32	1·70	2·47	2·11	·27	·75
Soluble salts .	1·21	1·44	·68	·88	1·95	1·61	1·86	1·35	1·30	5·92
Insoluble . . .	·92	·23	·56	2·01	·63	·54	·16	·03	..	·13
Water	4·32	4·36	4·10	3·04	5·34	5·52	5·60	9·96	3·88	5·07
	100·00	100·00	100·00	100·00	100·00	100·00	100·00	100·00	100·00	100·00
Colour, D. S. Cane sugar obtainable .	7	8	8	6	6	7	9		13	10
	75·8	72·5	76·7	76·3	57·5	50·4	53·8	51·9	87·5	57·9

The total amount of cane sugar in any given lot of the raw material is of prime importance. On it depends the quantity of refined sugar which it is possible to produce from the raw sugar in question. Next in importance is the amount of fruit sugar as well as of soluble saline matter (soluble salts), both of which to a great extent retard or prevent the crystallisation of the cane sugar. The amount and nature of the extractive matter containing the colouring principles determine the amount of animal charcoal, whether greater or less, which it is necessary to use in the refining process, as well as the extent to which, and the manner in which, the charcoal will thereby be deteriorated. The amount and nature of the insoluble substances present in the raw sugar is also an important point to be considered. When present to any great extent, difficulty is experienced in washing. Moreover, in

some cases it is exceedingly troublesome to filter—the coarse particles, on the one hand, choking up the filter cloth, whilst the finer particles pass through the filter cloth, to be intercepted by the animal charcoal, the decolorising properties and other functions of which it greatly deteriorates. Some varieties of East Indian sugar are peculiarly subject to this defect.

The object, therefore, which each refiner has in view is to never have to work but upon one and the same species of sugar. The work is therefore arranged accordingly, and is never altered, so that the workmen know what they have to do, and could do it blindly. They are thus always sure of bringing their refined sugar to the same percentage by following the instructions which have been given to them. Consequently the refiner should endeavour to regulate his work according to a product of predetermined quality, so that the routine of this work should always be the same and no chance of error may be allowed to creep into the manipulations; for there is nothing more dangerous in an establishment comprising a large number of workmen than to keep incessantly changing the method of working. If it does not give rise to mistakes, there is inevitable vexation, loss of time and money.

But it is only possible, in the case of small establishments, to always treat the same material, except in Russia, where the refiner, being the only buyer of native sugar, can lay down the law to the factories and require them not to deliver any but extra-white sugar.

The large sugar refineries of Paris, which work up enormous quantities of sugar, cannot therefore choose and pick, and are forced to take all as they come. They must therefore regulate the quality of the sugar which they send to the melting pans, so that afterwards the working of the factory may always be the same.

The refineries therefore consist of two distinct factories or departments: (1) That in which the sugars are sampled and blended so as to make mixings of a predetermined yield from them; and (2) the refinery, properly so called, where these mixtures are melted and undergo further treatment.

WAREHOUSES

The sugar refinery.—Sugars are bought in immense quantities and piled in vast warehouses, where they are classified—cane sugar on the one hand, beet sugar on the other. All these sugars are analysed so as to determine their percentage of sugar, ash, alkalinity, or acidity.

Blending the alkaline beet sugar with the acid cane sugar to produce a neutral mixture.—Beet sugar is alkaline, cane sugar is acid. This double property is at once utilised to produce a neutral sugar by mixing cane and beet sugar in the right proportions.

Again, if there be in stock white sugar and brown sugar, and if

the work of the refining be regulated on the treatment of sugar as titrating 88°, the white and brown sugars are mixed in the requisite proportions to produce 88°. All this, which appears very simple, nevertheless requires a profound knowledge of the nature of sugars, their origin, and so on; but it is a question of every man to his trade, and the expert is trained both by science and practice.

Saline impurities.—However, things are not quite so simple as all that. It is always desirable to remelt as pure sugars as possible. The most dangerous impurities are the saline matters, and these are present in the molasses which soil the sugars. If the raw sugars were freed from these molasses, products would be obtained ready for treatment containing much less salts, and the yield on refining would be greater.

Preliminary turbinizing.—The first treatment to which brown sugars of a certain grade are subjected is to free them by turbinizing them from the molasses which they contain.

Therefore the first department which is met with in a sugar refinery is a vast installation of turbines, which is of greater and greater importance according as it is decided to raise the sugars to be treated to a greater and greater degree of purity.

The raw sugar as it comes to the factory is first mixed, sieved if its nature will allow, and introduced into the turbines, equalising the charge as much as possible. Then it is clarified by a blast of steam, until the desired tint has been obtained by the expulsion of the molasses which discoloured the crystals.

Some refineries make quite an industry of this turbinizing. In fact, certain "seconds" clarified with sugar liquor become quite white when turbinized. Then this kind of sugar, when well sifted and mixed with ultramarine blue, is turbinized under those conditions for a long enough time for the crystals to amalgamate together; then the cakes, which are taken from the drum, are crushed and delivered for consumption as *granulated*.

This special refining is cheap and profitable, because the sugar which it produces is endowed with the same properties as the refined article. Moreover, the molasses which it produces yield, on reboiling, sugars which are melted and refined in the ordinary way. In Italy, where granulated sugars are easily sold, several large establishments work in this way, with so much more profit, on account of the duty on sugars being paid according to the tint, and that it is easy to procure very rich highly-coloured sugars which turbine very well. All countries which pay the sugar duty by the tint are similarly situated. In French factories the turbinized sugars, brought to the desired strength, are mixed. The sugar is shot from the sacks into a large hopper in the right proportions, white, brown, beet, or cane, to obtain the desired type. All these are mixed and melted. This preparatory work is the only one that requires some attention from

the refinery manager, who, aided by the laboratory and his special knowledge of sugars of different origin, ought to always maintain a constant supervision on all the operations which occur in this first stage of the process.

Solution or "blowing-up."—But where the special initial turbine is not in vogue, the first stage in the sugar-refining process consists in dissolving the raw sugar in water, so as to obtain a thick solution or syrup. The effect of different qualities of water—hard, soft, etc.—on the char will be adverted to further on.

Open-fire system of heating in early days.—In the early days of the sugar industry this was effected by heating the sugar and water in the right proportions over an open fire—a method long since abandoned, a steam heat being adopted instead.

Steam heat.—Its effect on sugar solutions.—By the use of steam as a source of heat, all risk of burning the sugar is avoided. Steam heat, however, is not without an injurious action on sugar, similar to that of burning by direct heat. The extent of this injurious action depends principally on the pressure of the steam used; that is to say, if it be used to heat the saccharine solution in the form of open or free steam, that is, by being injected into the liquid through an open pipe.

Two methods of applying steam for dissolving sugar have been successively in vogue:—

Naked steam.—Formerly the sugar and water were exposed to the action of open steam, that is, to the action of naked jets of steam injected into it from a series of perforated tubes of iron or copper, ramifying below a false bottom placed across a rectangular metal tank. Owing to the violent agitation and deafening blowing noise produced by the condensation and escaping of the steam, the name of "*blowing-up pan*" was applied to the tank in which the process was conducted, and the appropriate term of "*blowing-up*" was applied to the process itself. The action of the open steam dissolves the sugar very rapidly, the steam jet not only supplying the source of heat, but also the means of so agitating the liquid as to keep it in continual motion, little or no stirring being required.

Applying heat by means of steam jackets and close steam coils.—It was soon, however, found in actual practice, that, when naked steam was brought into contact with a solution of sugar in the manner described, it is much more injurious than when the solution, contained in a suitable vessel, is heated by applying steam to the outside of the vessel, *e.g.*, as in what is known as a steam-jacketed pan. The old-fashioned blow-up pans therefore were eventually done away with, in favour of vessels constructed on the steam-jacket principle, or of vessels heated by means of a close steam coil (Fig. 80, A, p. 374).

In sugar refining as it was practised extensively in Greenock, the different floors of a sugar house, generally six or seven, were arranged

so that in the process the sugar passed down from one to the other. The sugar was accordingly hoisted to the top storey, where it was emptied out of the hogsheads, boxes, baskets, or bags in which it had been imported, and then shovelled through holes in the floor into the blow-up pans, which are situated in the floor underneath. The blow-up pans are cast-iron vessels, some 4 or 5 feet deep by 6 to 10 feet in diameter. A pan of 10 feet deep diameter will dissolve in the necessary quantity of water about 8 or 10 tons of raw sugar. At a little distance from the real bottom of the pan is a false bottom perforated with holes. The sugar rests on this false bottom until dissolved. Between the false and the real bottom a series of steam pipes are laid. The false bottom prevents the solid sugar coming in that condition in direct contact with the steam pipes, and being partially burnt or decolorised. A mechanical agitator, fitted with blades, is made to revolve by suitable gearing both above and below the false bottom, so as to keep the liquid continually in movement. The operation is commenced by running into the pan a sufficiency of water or thin liquor. Here, it may be remarked that the dissolving of the sugar in the upper floor of a refinery, owing to the great height, is more practicable in localities where a good natural supply and pressure of water exists. Where there is but a feeble pressure of water, it may be better to dissolve the sugar on the ground floor and pump it up by means of an "air vessel" or "egg" or *montejus* to the top floor, to undergo the other stages of refining in its descent, as shown in Fig. 82.

In any case, the water or thin liquor having been run into the pan in sufficient quantity, the steam is turned on to the steam coil, and the sugar shovelled in as fast as it can be emptied out of the casks. The filling of the pan takes about half an hour, and the liquid should by that time have attained a density of 28° Baumé (45° Twaddell; sp. gr. 1.225), and the temperature as near as may be 180° F. (82° C.). The liquid then contains about 2 parts of raw sugar to 1 of water. As regards the density or specific gravity of the liquid, there may be considerable variation. In fact, the operation of effecting solution—or "blowing-up"—is not applied to weighed or measured portions of sugar and water, but portions of each are run in as the operator may deem necessary, his guide throughout being the saccharimeter of Baumé. The sugar to be employed is scattered loosely over the floor, and the workmen throw it into the solution tank like so much earth.

No two refiners are exactly agreed as to the exact density. Perhaps that which we have just given may be the best. Horsin-Deon gives 37° to 39° B. (sp. gr. 1.34 to 1.36).

Hitherto only a solution of raw sugar in water has been dealt with, and during the heating of this solution a scum rises to the surface, which is skimmed off by almost flat perforated ladles, but

the amount of flocculent insoluble material skimmed off in this way is really very trifling. But be that as it may, this is the simple manner in which the solution was effected in most of the old Clyde refineries. In other localities, however, the liquid is subjected to different kinds of treatment in the "*blow up*," so as either to remove a portion of the colouring matter or to neutralise any traces of acid in the sugar.

This acid may, it is said, be either pre-existent in the sugar (malic acid, etc., p. 282), or be developed during the stay of the sugar in the hold of the ship, or in the warehouse (acetic acid, lactic acid).

Again, the liquid may be treated in the pan with a view to facilitate subsequent filtration, so as to produce a perfectly clear and bright liquid.

In order to neutralise the acidity when working with cane sugar alone without any admixture with beet, it is a common practice to add a sufficiency of milk of lime or lime water for the purpose. Although this process is not a general one even in some of the most skilfully managed refineries, it may, however, be regarded as an advantageous one in many instances. It must not, however, be used in excessive quantities, otherwise it will darken the colour of the liquid, and thus throw extra work on the animal-charcoal filters. The lime is said to be employed for the purpose of correcting acidity, but this explanation is not altogether correct. It is not complete. It has been pointed out that if only the mere neutralisation of the acidity were required, that is to say, if we use the term acidity in the ordinary acceptation of the term, chalk should succeed as well as lime, and its employment would be free from many of the risks of injury to the sugar with which the use of lime is attended. Again, it has been suggested that the existence of an acid in unfermented sugar is hypothetical, and, moreover, if the antacid quality of lime were especially desired, lime water, containing as it does so little of that earth, would be a very inefficient medium of administration. The real function of lime water in sugar refining would appear to be that its lime combines with the various colouring principles existing in raw sugar to produce a flocculent precipitable substance. Again, bullock's blood, in virtue of its coagulating by heat, was formerly added to the blow-up pan, and was technically known as *spice*. When the bullock's blood coagulated, it enveloped, in the albumen of the serum, the flocculent precipitable substance produced by the action of the lime on the colouring matter of the sugar, as well as any other impurities present; and the whole floating to the top of the liquid in the form of scum, which is easily removed, the solution of sugar was more or less clarified. The coagulated albumen, in virtue of its affinity for colouring principles, was said to exert of itself a bleaching action on the solution of sugar. As more cleanly substitutes for blood, solid albumen obtained by appropriately evaporating white of eggs, or the serum of

blood at a low temperature, has been used. But in any case these substances render the syrups very impure, and are now but seldom used. Horsin-Deon, however, still mentions $1\frac{1}{2}$ to 2 per cent. of ox blood, and 2 to 4 per cent. of fine char, as well as sometimes a little caustic baryta, to precipitate certain organic and mineral matters. Amongst the various substances which have been suggested as substitutes for blood, a mixture of sulphate of alumina and lime, the ingredients of which react on each other to form a gelatinous precipitate, may be mentioned; but this mixture is objectionable, because it introduces calcic sulphate, soluble phosphate of lime, and soluble phosphates of alumina and lime. Spencer, in his report on the diffusion *versus* milling work at Magnolia, U.S.A., says: "I have frequently objected to the use of superphosphate of lime and alumina. These superphosphates usually contain an excess of the sulphuric acid used in their manufacture, and the stock at Magnolia was no exception to the rule. It is difficult to find any advantage arising from the use of these superphosphates." According to Wallace, the greatest objection to these mixtures is the danger of the common workmen, who are necessarily entrusted with the application of them, using excess of one or another, and thus rendering the liquor acid or alkaline, and so doing more harm than good—more especially as it would not appear to be indispensably necessary to add anything to the liquor, as many refiners of the first rank use no chemical reagent.

Animal-charcoal dust is sometimes introduced into the blow-up pan, so as to decolorise, or partially decolorise, the sugar solution. Provided the charcoal be new, that is, has not been used before in sugar refining, it produces a good result; but, owing to its getting contaminated with the impurities in the sugar, it can only be used once.

An abundant scum is formed, which is continuously broken by the agitator. The whole is then passed through the Taylor filters (Figs. 77 and 82).

Sometimes clarification is effected in other tanks (Fig. 82, A) from those in which the sugar is melted. When the mixture of syrup, blood, black, and baryta is effected, this liquid is pumped by a *montejus* (Fig. 82, H) or a pump into a closed pan (Fig. 82, I), where a slight vacuum is made which induces boiling and agitates the mixture. The vacuum is broken by and by, the temperature is raised to 105° C. so as to complete the coagulation of the blood albumen, after which it is allowed to flow into the bag filters (Fig. 82, N).

Filtration, therefore, involves two operations—(A) Passage through Taylor's filter bags; (B) through bone char.

A.—REMOVAL OF SUSPENDED IMPURITIES BY BAG FILTRATION—
TAYLOR'S FILTERS

The clarified syrup must be freed from any suspended matter which may be present in it—coagulated blood, black, and precipitates of all kinds. It therefore undergoes a filtration process through the Taylor filters. These filters are of two kinds: either the liquid enters by the inside, depositing its impurities within the bags,—or the bags, kept stretched by an inside lining of osiers and packed in tight cases, dip into the liquid, which passes through them, leaving all the impurities behind in the case. No matter which of the two methods be adopted, the filtered syrup should be quite limpid.

When the mixture of sugar and water has become thoroughly dissolved, and is of the appropriate density, and when the coarser impurities which have risen to the top of the liquid in the form of scum have been skimmed off, a sluice or tap M is opened in the bottom of the pan (Fig. 82), and the fluid is allowed to run into a shallow tank or tray (Fig. 77, *a* and Fig. 82, N), to the bottom of which a very ingenious system of filtration is adjusted. In the bottom of the shallow tank *a* (Fig. 77) there are numerous apertures *o, o, o, o*, each of which is cut with a screw into the form of a female screw. Into each of these a brass filter bell *b*, terminating in a tube in the form of a male screw, is tightly screwed.

To each of the filter bells there is securely tied at *d* a filter bag *c*, of twilled cotton, in the shape of a pillow-case or bolster, of about 2 feet in diameter, and each of these bags is thrust into another bag *s*, of coarse canvas of about 6 inches in diameter. The outside bag is very appropriately termed a sheath. These bags are some 6 or 8 feet long, and are fastened, as described, to the number of 200 or so to the bottom of the shallow tank in question;—the whole being surrounded by the sides of an iron box (Fig. 82, N), so that the liquid may be kept hot by means of steam introduced for the purpose.

Peculiar adaptability of the process to the end in view.—It will thus be seen that this filtering arrangement is well adapted to provide a wide superficial area in a compartment of comparatively small cubic content—a state of matters which it is highly desirable to accomplish in a sugar refinery where operations are conducted on so large a scale in working premises of not unlimited dimensions. The buildings of a sugar refinery would be spread over too wide a superficial area of ground, if space were not utilised to its fullest extent without cramping. The above system of filtration is therefore a very happy and effective illustration of the adjustment of means to an end. Not only is it economical in space, but it is about as effective a means as can be imagined of what might be called automatic filtration, in contradistinction to filtration through filter presses. Other mechanical filters are described on pp. 170–175.

Points which require attention in bag filtration.—Several points, many of which will suggest themselves to the practical man, require to be attended to during filtration. The most important point is to fill the bags with the sugar solution in as short a time as possible.

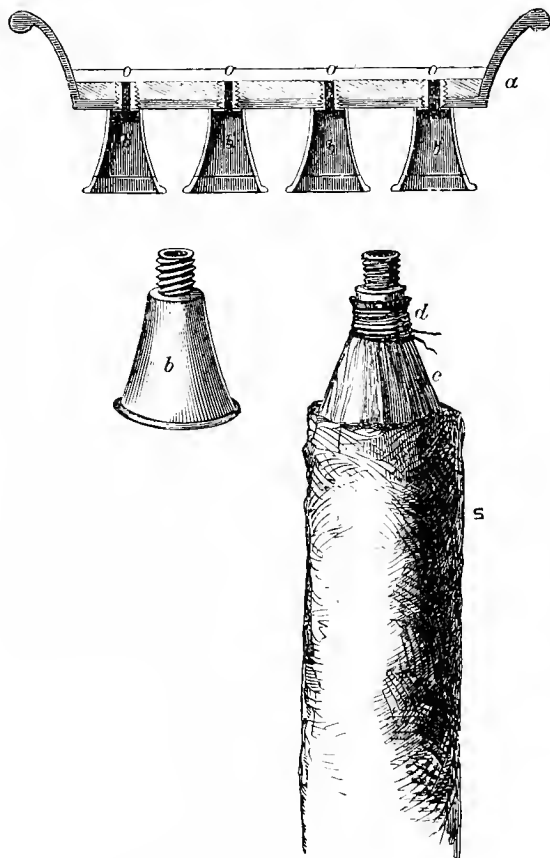


FIG. 77.—Taylor's filter bags.

Unless the bags are completely full the filtrate comes away foul. Moreover, the filtering arrangement (Fig. 82, N) must be warmed up to the right temperature before the liquid is run off from the blow-up pan. This, as already indicated, is accomplished in a very simple but very effective manner, namely, by suspending the filter bags in a box or

chamber, into which steam jets may be turned on or turned off at will. The filtered liquid as it comes from the filtered bags is sampled and examined from time to time by a workman. He takes a sample in a test-glass, and examines it by transmitted light to see whether it is perfectly bright. The brightness of the liquid is a very important point, as any turbidity injures the quality of the sugar-loaves to be made from it in the subsequent operations. Until the filtered fluid comes away perfectly clear and bright it is run into a receiver called the foul-liquor tank, from which it passes back to the blow-up pan.

It has been suggested to replace the Taylor filters by filter presses, which answer very well if the filter cloth has been well selected. But sometimes it is necessary to add solid matters in suspension to counteract the gluey nature of the muddy residues left on the filter cloths, which would otherwise choke up its pores.

This sludge, called refinery black, forms an excellent manure containing the phosphate in the black, the nitrogen of the blood, the sulphuric acid and salts, and the whole of the other organic matter. This manure is deservedly held in high esteem by farmers intelligent enough not to object to its black colour.

B.—DECOLORISING THE SOLUTION OF RAW SUGAR—REMOVAL OF DISSOLVED IMPURITIES

1. *By animal charcoal.*—When the maximum degree of brightness and freedom from turbidity have been obtained, if the filtered liquid be examined it will be found that, however bright and clear the liquid may be, its colour will be dark, not unlike that of port wine. The next process, therefore, after having got the solution clear and bright, is to get rid of the colouring matter,—to decolorise it, in fact, or to bleach it. This is accomplished more or less effectually by passing it through or bringing it into contact with more or less finely ground animal charcoal, or bone black as it is sometimes termed. This substance is the same as that sold to artists under the misleading name of ivory black.

The excellent decolorising properties of animal charcoal may very well be illustrated, or demonstrated, by agitating a small quantity of port wine, tincture of litmus, cochineal, or any other coloured solution with it. Or, better still, if the coloured solution be passed through a layer of bone black, the filtrate will be more or less completely decolorised.

The use of other charcoals impracticable.—Although wood, etc., charcoal also possesses decolorising properties, animal charcoal has been found practically to be most suitable for the purposes of the sugar refiner. Indeed, many varieties of charcoal have been

tried, and, moreover, some of these are much more energetic and effective as decolorisers than bone black. But, owing to other and objectionable properties, it has not been found practicable to render this superiority in decolorising properties available in sugar refining.

Peculiar adaptability of animal charcoal.—As a matter of fact, animal charcoal is alone endowed with that essential aggregation and combination of characteristics and properties which is required in any substance which has to effectively fulfil the rôle of a decoloriser of sugar solutions. Such a substance must be free from soluble inorganic salts, that is, from salts which readily dissolve in water. Neither must it contain any very appreciable quantity of sulphate or carbonate of lime. Moreover, whatever decolorising agent may be used, its specific gravity must be at least so great that it will sink freely in the densest of sugar liquors, whilst at the same time it must be extremely porous; and not only so, but it must be hard and coherent enough to bear handling and the rough usage to which it is subjected in being carted and shovelled about and reburned every other fourth day for several years in succession. No charcoal fulfils these many and varied requirements in such a marked manner and to so great an extent as animal charcoal. By animal charcoal is meant the charcoal made from bones. Charcoal made from such substances as leather waste might be regarded as animal charcoal. Yet although the charcoal is highly esteemed in the metallurgy of the noble metals, yet for many reasons it is unsuitable for sugar refining. The practice of animal-charcoal manufacturers and sugar refiners of quenching the red-hot charcoal as it comes from the kilns with water is greatly to be deprecated, as being highly injurious to the decolorising properties of the char. The water acting on the red-hot nitrogenous carbon evolves ammonia, and thus diminishes the proportion of the nitrogen, which is supposed to play such an important rôle. Some animal-charcoal makers strive, for the sake of a little extra profit, to distil off as much ammonia from the bones as possible, so as to secure a larger yield of sulphate of ammonia, and it has even been suggested to inject superheated steam into the retorts. No doubt, this innovation would yield a larger quantity of sulphate of ammonia per ton of bones burned, but it would inevitably deteriorate the decolorising properties of the resulting charcoal.

Selection of good quality charcoal.—This is a most important point. New animal charcoal—that is, freshly calcined bones—consists essentially of carbon, tribasic phosphate of lime ($\text{Ca}_3\text{P}_2\text{O}_8$), and carbonate of lime (CaCO_3), together with small proportions of a few other substances. Its composition is not definite, varying somewhat with the source of the bones.

The following analyses by Wallace will, however, give some idea of its general composition.

LXVIII.—ANALYSES OF NEW ANIMAL CHARCOAL.*

Dry.	A.	B.	C.
	Per Cent.	Per Cent.	Per Cent.
Carbon (nitrogenous)	9·71	7·64	10·37
Calcic phosphate .	80·48	84·05	78·70
Calcic carbonate	8·82	7·61	8·05
Calcic sulphate	0·31	0·20	0·53
Alkaline salts	0·30	0·25	0·58
Ferric oxide	0·12	0·15	0·21
Silicious matters	0·23	0·10	1·56
	100·00	100·00	100·00

* Analyses of old and used chars are given on p. 354.

Nitrogen, etc., in animal charcoal.—As there is a very appreciable quantity of nitrogen and a small amount of hydrogen in the so-called carbon of animal charcoal, it is far from being pure. The proportions of both these elementary bodies present in any given char depend upon the temperature to which the charcoal has been exposed in the charcoal burners. As a general rule, the nitrogen amounts to about one-tenth part of the total carbonaceous matter, but occasionally it is even more. The amount of hydrogen in well-burnt animal charcoal is very small indeed. Dr. Wallace quotes a case in which a new char only contained 0·034 per cent. Char which has been used over and over again in sugar refining and reburnt in the interval does not contain so much nitrogen, and the proportion would appear to diminish in a direct ratio with the number of times the charcoal has been reburnt and re-used in sugar refining. In a not very old charcoal it was found to be as low as 0·3 per cent., and no doubt in still older charcoal would be much less.

Function of the nitrogenous carbon in animal charcoal.—The whole rationale of the decolorising effects of animal charcoal is shrouded in mystery, and no point is more obscure than the rôle of the nitrogenous carbon. That it plays an important part, there can be no manner of doubt. Some authorities even go so far as to assert that it is an essential constituent of animal charcoal, and that no char which does not contain an appreciable quantity of nitrogen is a good decolorising agent. It has been pointed out that

wood charcoal, for example, although exceedingly permeable and an excellent absorber of gases, is a very poor decolorising agent, and is practically useless for the purposes of the sugar refiner.

The injurious action of the presence of free ammonia and of ammonium sulphide in new char.—Traces of free ammonia are always present in new charcoal as originally received from the animal-charcoal maker. The proportion, however, is exceedingly small in one particular case, being as low as 0.011 per cent. The evil action of even this small quantity and of small accompanying traces of sulphide of ammonium is readily perceived in the sugar liquors which have been run through new charcoal, which should never be used until after it has been well washed and reburned. In addition to the above injurious substances, new charcoal always contains a small quantity of calcium sulphide, and gives off sulphuretted hydrogen when acted on by an acid, and even when only moistened with water. In one instance Wallace found 0.08 per cent. of hydric sulphide in a sample of new char after treatment with acid.

Presence of interstitial or occluded gases in new and old animal charcoal.—Both new and old animal charcoals retain within their pores appreciable quantities of gases, which escape when tanks containing them are filled with liquor. Moreover, these gases have been known to explode at the approach of a light to the top of the tank.

2. *Substitutes for animal charcoal.*—Attempts have been made from time to time to mix clay with some one or other form of carbon, so as to produce a mixture which would compete with animal charcoal. The charcoal from certain sea weeds is said to rival that from bone, but it does not fulfil all the requirements indispensable to a sugar decoloriser.

3. *Basic acetate of lead and sulphurous acid (Scoffern's process).*—Other purifying and decolorising agents, besides lime, blood, and animal charcoal, have been suggested from time to time; amongst these, mention must be made of Scoffern's basic acetate of lead process. The prime object of sugar refining is the removal of impurities and colouring principles. Not many, if any, reagents are more efficient in doing so than the basic acetate of lead. Although this fact was well known to chemists, the process was not available owing to the difficulty experienced in removing the excess of the poisonous basic acetate of lead without simultaneously injuring the sugar. Dr. Scoffern, however, accomplished this in a very ingenious and effective manner by forcing sulphurous acid gas through the lead-treated sugar solution by means of a force-pump. By this means every portion of the lead was said to have been removed.

Professor Brande's opinion of Scoffern's process.—In regard to this process the late Professor Brande said: "I do not appear here as the advocate of Dr. Scoffern's process or any other, but it is

only just to say that the opinion I gave two years ago in favour of the efficiency of this process and of its absolute safety, when carefully and skilfully conducted, has been fully borne out by my experience since that time. I believe the means of extracting the last portions of lead by Dr. Scoffern's process are infallible, and that they involve no greater amount of difficulty or skill than may be commanded by every sugar manufacturer who chooses to devote adequate attention to the subject." However authoritative Dr. Brande's opinion may have been, and however entitled it may have been to respect, the Government of the day, in deference to public opinion—perhaps prejudiced—denounced the process.

4. *Sulphurous acid*.—Sulphurous acid itself has been repeatedly tried as a sugar-bleaching agent, and, as was long ago pointed out by Dr. Wallace, processes are published every two or three years, in which its use is advocated—each writer apparently unaware that his results are well known. At its best, sulphurous acid, according to Wallace, only removes about three-fourths of the colouring matter, and the liquor requires to be treated with charcoal, just as much as if sulphurous acid had not been used. Again, Wallace points out that although it is true that sulphurous acid does not alter cane sugar, like most other acids, yet its liability to change into sulphuric acid involves the risk of introducing a dangerous element; and although the acid itself may be neutralised by lime, the resultant compound formed by the combination of the lime with the sulphuric acid, namely gypsum, is very injurious to the charcoal (by which it is intercepted) in subsequent operations, and renders it less efficient after reburning, if not even injurious owing to the formation of calcium sulphide.

5. *Decolorisation by ozone*.—It has not been found practicable to decolorise saccharine liquors by ozone, and the opinion has been expressed that, even if found to be economical in bleaching the colour, it would not be possible to dispense altogether with animal charcoal. Moreover, it has been anticipated that its oxidising action would prove troublesome. Beanes, however, claimed that ozonised air passed through a coloured syrup for three hours exerts a decolorising action as great as that obtained by leaving the sugar in contact with animal charcoal for twenty-four hours.

6. *Defecation of cane sugar by the double carbonatation process as applied to the sugar beet*.—The double carbonatation process, so successfully applied on the Continent to the refining of beetroot sugar, has been but little applied on the large scale to the process of refining raw cane sugar. Moreover, it is not anticipated that its application would be attended with any great advantage. As a matter of fact, the sugar solution has to be rather dilute before this process can be applied, and in consequence it would have to be boiled down before being passed through the charcoal, as it is not by any means

a substitute for charcoal, but is only supplementary to the action of the latter. Experiments in this direction tended to show that the colour of the cane-sugar solution was little or at all improved. But, on the other hand, the liquors obtained are particularly clear and bright. In certain special cases, however, such as in purifying the washings of animal charcoal, which are highly impure, indeed, and especially troublesome, as well as bag-filter washings and the other impure liquors obtained during the various stages of the refining process, the double carbonatation process might be advantageously adopted in the refining of cane sugar.

Wallace, indeed, recommends that such impure saccharine liquors as bag-filter and char washings and low syrups, much contaminated with salts, should all be purified by precipitating the sugar as a sucrate of lime, and then separating the base by carbonic acid gas; or, in the case of baryta, by sulphurous acid gas. In this connection he suggests as an excellent arrangement, to have connected with every large refinery a smaller one immediately adjoining it, where all impure products are treated and worked up apart from the more pure sugars made in the refinery proper. (See *sucrate annexe*, Molasses, pp. 268-270.)

DECOLORISING SOLUTIONS OF RAW CANE SUGAR BY FILTRATION THROUGH ANIMAL CHARCOAL

The solution of raw sugar, after having been rendered clear, limpid, and transparent by being passed through the bag filters (Fig. 77), is run into iron tanks or cisterns filled with bone black (Fig. 41, p. 160, and Fig. 80, O, p. 374). These tanks vary greatly in size and shape; some of them are square and shallow, whilst others are of great depth, attaining in some cases a depth of 40 to 60 feet. The form most generally used is that of a circular iron cylinder, say about 9 feet in diameter and 16 feet deep. Such a cylinder will contain about from 20 to 25 tons of charcoal, according to its density. This cylinder is packed with the granulated charcoal, which rests on a false bottom of stout linen cloth supported on perforated wood-work. The charcoal generally extends from the false bottom upwards to within a certain distance, some two or three feet, of the top end of the cylinder. The remaining space serves as a reservoir for the sugar solution, which gradually percolates through the charcoal and is allowed to escape into a receiver. In some cases the cylinders are open at the top (Fig. 41), whilst in other cases they are closed as in the Clyde sugar refineries, except the necessary hole for the insertion of inlet and exit tubes and manholes for charging and discharging the animal charcoal, and cleaning the tank and filtering arrangement generally. The tanks are substantially built of wrought iron. They are, in fact, constructed to bear the pressure of a considerable volume of water, which may be applied when necessary so as to expedite filtration.

Amount of charcoal required to decolorise given weight of sugar.

—The weight of charcoal required for a given amount of sugar is very variable. It depends to a great extent, as has been pointed out by Wallace, on the greater or less abundance of water and on the price of coals, and whether or not the charcoal has to be sent to a distance for reburning. In the latter case, and where coal is dear and water scarce, the quantity of charcoal is reduced as far as practicable, but under more favourable circumstances the ratio should not be less than 25 cwts. of char to a ton of sugar.

Size or grist of the granulated charcoal.—This greatly depends on the dimensions and shape of the cisterns, but, where practicable, small-sized granules should be selected. Looking at the question from a theoretical point of view, the smaller the grist the better should be the result; but, as a matter of fact, the charcoal granules must be sufficiently large to leave a passage for the liquor to pass through it in something like a reasonable time. In actual practice, such a size as would pass through a sieve of twenty meshes to the inch, but would be kept back by one of thirty meshes to the inch, has been found to answer best.

The sugar liquor is run from the blow-up pans into the iron tanks filled with animal charcoal as described, where it is allowed to stand for some hours. It has been found by experience that the *most suitable temperature* for the charcoal to exercise its greatest decolorising effect on sugar is for the liquid to be run off from the blow-up pans at 180° F., whilst the charcoal cisterns should be kept at about 155° F., and never below 150° F. After the sugar solution has been left for a sufficiently long time in contact with the charcoal it is slowly run off from below, while more of the dark-coloured liquor automatically flows on to the top through the ball valve (Fig. 41), so that the tank is continually full. The liquor which comes away at first is not only bright, but is colourless as water. Gradually, however, as the process goes on, the liquid becomes more and more tinted, until after a certain time it becomes distinctly yellow or sherry colour. The sugar solution is then replaced by the syrup from a previous refine.

Charcoal washing.—The syrup is then washed out with hot water. This is an operation of some delicacy. The hot water is run in and allowed to percolate the mass until no appreciable trace of sugar can be found in the washings, that is, until the escaping fluid marks zero, or nearly so, on the saccharimeter—a sure sign that all, or nearly all, the sugar has been washed out of the charcoal. If this operation, therefore, has been skilfully conducted, little or no sugar will remain behind. The great object of the refiner is to remove a maximum of sugar with a minimum of water. The charcoal is now further washed with copious volumes of boiling water, then with cold water, after which it is taken from the

tanks and transferred to the kilns to be reburned. The minimum amount of water used in refining 100 tons of sugar is—

WATER USED IN REFINING 100 TONS OF SUGAR.

	Tons.
Dissolving	50
Washing to produce sweet washings, afterwards boiled down or used for dissolving	40
Extra washing to further purify char	125
	215, or 50,000 gallons.

Mr. Spencer, in his report on diffusion working *versus* milling working at Magnolia, says: "In making these grades of sugar, necessitating a double filtration of the syrup, double the loss was experienced in the bone-black room, due to the absorption of sugar by the char and the losses in the waste waters. The filters were open Dumonts (Fig. 41). Owing to the difficulty of properly washing the char without employing a very large quantity of water, the waste products contained considerable sugar, as the following table will show:—

LXIX.—LOSS OF SUGAR IN BONE BLACK AT MAGNOLIA.*

Date.	No.	Sucrose Per Cent.
Nov. 21	1	2·82
" 22	2	·91
" 22	3	1·12
" 22	4	·44
" 23	5	·73
" 24	6	·91

* The normal loss is given on p. 353.

These analyses showed an enormous loss in the filter-room. After November 24, orders were given the filter-men to wash their filters a very considerably longer time than they had been doing. Samples were frequently brought to the laboratory for examination, and whenever the proportion of sucrose exceeded ·50 the washing was continued. It is impossible to estimate the loss from this source, but it was certainly very large. It was impracticable to vary the mode of work in the bone-black room. The quantity of waste water was variously estimated at from 1000 to 1500 gallons per 24 hours; this would indicate losses ranging from less than 100 to nearly 300 lbs. of sugar per day, or from 0·5 to 1·5 lbs. per ton of cane. As there were no means of ascertaining the amount of waste water, these figures are a rough approximation of little value"

The *suspended* matter in raw sugar liquors, as already mentioned, is removed in the process of bag filtration. It is the objectionable *soluble* substances in the raw sugar liquors that are removed and retained by the animal charcoal. The first thing to be done, then, is to wash out the sugar (as already described) which has penetrated into the pores of the charcoal. But at this point a serious difficulty presents itself. The bulk of the impurities retained by the charcoal are far more soluble in the hot water used for washing than in the original strong raw sugar liquor. They are therefore partially removed in the wash-water. The impurities of the original raw sugar liquor are therefore concentrated in the weak saccharine liquor obtained by exhausting the char, by washing with *boiling* water to such an extent that it is far more impure than the original sugar liquor from which they had been extracted. Vegetable albumen, gum, salts of lime, potash, soda, and iron are eliminated from the char by washing, and are concentrated in the liquid along with but a small quantity of sugar. The usual way of working up this *sweet water*, which is extremely liable to fermentation, is to use it up in dissolving fresh raw sugar, but it is hardly possible to make a greater blunder in sugar refining. This has been aptly described as first taking dirt out and then putting it back in its place, to be again removed with the next accumulation. The free oxygen in the water was at one time said to act under the influence of the bone black upon the vegetable albumen and to change it into a ferment which quickly converts the sugar into lactic acid, and this acid dissolves from the charcoal both lime and iron. The charcoal washings are therefore very often sour and putrid and heavily charged with lime salts; and not only so, but they frequently emit a perceptible smell of sulphuretted hydrogen.

When the sugar has all been washed out of the char by prolonging the washing, as already described, until the char cedes but little more to the wash-water, the char is then drained, taken out of the cisterns, and transferred to the kilns (Fig. 43, p. 169).

The kind of kiln most generally used comprises a series of vertical cast-iron pipes about 5 inches in diameter, arranged in six rows of about ten pipes in each row. Three rows of these pipes are placed on each side of the furnace. They pass vertically through a brick chamber, and are kept at a red heat by furnaces underneath, the products of combustion being drawn off from the sides of the kiln. The pipes are open at the top, where they pierce the brickwork. Underneath they end below the fire chamber in flat sheet-iron prolongations, called cooling boxes, of the same length as the row of pipes to which they are attached, about 6 or 8 feet deep, and an inch or three-quarters of an inch wide, and cooled by simple contact with the surrounding air. In this way the temperature of the charcoal is reduced below the ignition-point before it comes

in contact with the superincumbent atmosphere. The wet char, as it is brought from the filtration tanks, is laid on the top of the kilns, and raked down into the pipes, in which it sinks gradually down as the burnt char falls into the cooling box underneath. From time to time, say every twenty minutes, the bottoms of the coolers are opened, and the char in them partially removed, in such proportion that it will have occupied six to eight hours in its passage through the pipes. The exact length of time will, of course, depend on the heat of the kiln, and different amounts should be drawn from each individual row of pipes, according to their proximity to the source of heat. If there be three rows of pipes, the row next the fire should be discharged in, say, five hours, the middle row in about seven and a half hours, and the back row in ten hours. These kilns economise fuel fairly well, but one of their great defects is that the wet mass of superincumbent charcoal forms an obstacle to the free passage of the steam and gases evolved from the simultaneous drying and carbonising of the vegetable impurities in the charcoal. As a matter of fact, four-fifths of the heat consumed in the kiln are spent in drying. It is therefore highly desirable to dry the charcoal more or less before running it into the pipes.

When the charcoal has sufficiently cooled, it is again run into the animal-charcoal tanks. The volumes of water capable of being absorbed and retained by the same quantity of old and new charcoal differ in a very decided manner indeed. For instance, if a funnel be filled with good, perfectly dry new charcoal, and water poured on to it as long as it is absorbed, it will be found to retain in its pores from 80 to 100 per cent., while old charcoal only absorbs from 30 to 45 per cent., according to the quality. Moreover, dry new charcoal does not become perceptibly wet unless at least 20 per cent. of water is added to it, while old charcoal becomes wet with even 5 per cent. It will thus be seen that the practical man need experience no difficulty in differentiating between old and new charcoal. Wallace quotes the results in the following table. A quantity of new char was exposed to a rather strong heat for an hour in a covered crucible, with the following results:—

LXX.—CONTRACTION AND CONSOLIDATION OF CHARCOAL IN HEATING.

	Cubic Feet per Ton.
New charcoal	48
After heating for one hour	43·2
After other four hours	38·0
After still another four hours	35·5

The charcoal thus lost in eleven hours as much of its porosity as it would by being worked in a sugar house for two years. Calcic phosphate is fusible at a great heat, but the temperature of a charcoal furnace is only sufficient to produce agglutination.

Differentiation between old and new charcoal.—New charcoal burnt till white has the appearance of little pieces of chalk, but old charcoal has the semivitreous compactness of porcelain or flint.

Changes in composition on repeated reburning and re-use of animal charcoal.—*Increase of carbon.*—*Evil effects.*—Old charcoal differs in composition from new charcoal. The proportion of carbon always increases, and if the kilns be perfectly tight this increase is quite rational. In this way the pores of the charcoal are gradually filled up with a deposit of carbon, due to the carbonisation of the organic matter extracted from the raw sugar liquor and retained by the charcoal. This ever-increasing deposit of carbon is an unmitigated evil in sugar refining. Every effort should be concentrated to abate it by thoroughly washing the charcoal by boiling hot water before reburning.

Decrease of carbon.—The proportion of carbon in some refineries would not appear to augment, whilst in others it rapidly diminishes, so much so that in exceptional cases it eventually does not exceed more than 2 or 3 per cent.

Due to access of air.—Such a great loss of carbon as this can only be due to faulty arrangements at the kilns. Air must find access to the charcoal in the pipes either through flaws in them or some way or other, but with ordinary care this should easily be prevented, as it is by no means an easy task to burn off the carbon from the charcoal that has been used for sugar-refining purposes, so as to produce a white bone ash available for making a white or grey coloured superphosphate after treatment with sulphuric acid. The farmer shrugs his shoulders, or perhaps expresses his disgust more forcibly, at a manure of the colour of boot-blackening. Be this as it may, care must be taken to prevent air getting access to the hot char. The pipes and cooler boxes must be kept as air-tight as possible.

Loss of carbon by overheating.—On the other hand, loss of carbon may very well take place by the pipes getting overheated to such an extent as to cause a reaction between the carbon and the elements of water, with the resultant formation of carbonic oxide and marsh gas. However, if the pipes and cooling boxes are air-tight, and the charcoal be not subjected to any undue heat, the carbon must perforce increase rapidly if all, or nearly all, the organic matter absorbed from the sugar liquor be not regularly and systematically washed out of the charcoal each time it is used before reburning.

Removal of mineral salts from the charcoal.—One of the great benefits derived from copious washing of the charcoal is the removal of mineral salts absorbed by the charcoal from the raw sugar

liquor. All raw sugars invariably contain a certain proportion of mineral salts, which may vary from $\frac{1}{2}$ to about 1 per cent. in the general run of cane sugars, from 1 to about 2 per cent. in syrup sugars, and in beet sugars from $1\frac{1}{2}$ to 7 per cent. It must be observed that highly soluble salts, such as those of potassium, have little or no effect upon the charcoal. They are, however, a source of great annoyance to the sugar refiner, by accumulating in the syrups, from which they can, however, be readily eliminated by a very elegant process patented by Messrs. Newlands, which will be described later on. The more imperfectly or but slightly soluble salts exert a very injurious effect upon the charcoal, unless removed by copious and thorough washing. The most objectionable in this respect is calcium sulphate, a salt only but very little soluble in water. This salt is readily abstracted from the sugar solution by the charcoal, and, moreover, it can only be eliminated therefrom by copious washing. A very peculiar point about the absorption of calcium sulphate by charcoal is that it is only absorbed from the strong sugar liquor, but is ceded by the charcoal to the wash-water, in which it is carried away to such an extent that an abundant crop, not of sugar crystals but of gypsum, is often obtained in boiling down weak char washings. (See Table XXXI. p. 118.)

Bad effect of permanently hard water when used to wash charcoal.—In the case of a permanently hard water containing much sulphate of lime in solution—saturated with it, as it were—it is almost altogether impracticable, if not quite impossible, to effectively wash the charcoal, and, as a matter of fact, instances are not unknown of charcoals contaminated in this way with sulphate of lime to the extent of $2\frac{1}{2}$ per cent.

The use of lime involves treating the char with acid.—Again, in those factories where lime is used in clarifying the juice, especially in beetroot factories where it is employed in such quantities, the pores of the charcoal quickly become clogged or choked up with carbonate of lime to such an extent as to destroy its beneficial effect on the raw sugar liquor. The only way to restore some of its pristine properties to the charcoal is to treat it with an acid to remove the excess of carbonate of lime (p. 167).

Influence of repeated use and reburning on the density, volume, and porosity of animal charcoal.—As charcoal ages from repeated use and reburning, its volume gradually contracts or shrinks to a very appreciable extent, so as to diminish or even completely obliterate the pores. According to Wallace, the space occupied by a ton of new charcoal when dry is usually about 50 cubic feet, but after being a few months in use it is reduced to 40, and so it goes on shrinking until it reaches 28 cubic feet, which, out of 400 samples examined by Wallace, was the most compact. It must not, however, be imagined that the specific gravity of the charcoal is in any way

increased. The density, in fact, of old and new charcoal differs but little, as will be seen from the following table:—

LXXI.—OLD AND NEW CHARCOAL—RATIO OF BULK TO GRAVITY (WALLACE).

	Cubic Feet Per Ton.	Specific Gravity.
New Charcoal . . .	50·6	2·822
Old Charcoal . . .	35·0	2·857

According to Wallace, the heat to which the char is subjected produces a semifusion of the calcic phosphate, which is the most abundant constituent of charcoal, and causes a shrinking in the bulk of the particles. This, however, can hardly be the case, because any actual shrinking of the substance of the charcoal, as distinguished from the coalescence of the particles to form, as it were, one united whole, must of necessity increase the density.

Amount of charcoal per ton of sugar.—The exact amount depends to a great extent upon the quality of the sugar to be refined, and also upon the quality of the charcoal. It stands to reason that the smaller the amount of charcoal used consistent with effectual working, the better. The larger the amount of charcoal used, the greater is the loss of sugar, but it also involves the production of abnormally large quantities of weak, highly impure, wash-water. As already mentioned, 25 cwts. of charcoal to 1 ton of raw sugar are quite sufficient if the sugar be of good quality. In the case of fine sugars, equal quantities of sugar and charcoal suffice. One of the greatest mistakes that can be made in sugar refining is to imagine that a large quantity of bad or spent charcoal will do the same work as the ordinary amount of good charcoal. In the first place, not only does it occupy a great deal more space and thus curtail the yield of refined sugar, but, however large may be the quantity of charcoal used, it does not accomplish the end in view nearly so well. Moreover, it yields an overwhelming bulk of sweet water, *i.e.* charcoal washings. Working on the large scale, it has been found in actual practice to be impossible to eliminate all the sugar from the charcoal by washing, so that it would be worth while to recover the sugar by evaporation, concentration, and crystallisation. Again, there is a loss of 0·75 per cent. of sugar on the charcoal used, *i.e.*, for every 100 lbs. of charcoal employed, $\frac{3}{4}$ lb. of sugar is lost. Accordingly, if an amount of charcoal equal to that of the sugar be used, the loss will be 0·75 per cent., whilst, if the amount of charcoal be double that of the sugar, the loss will be $1\frac{1}{2}$ per cent. of sugar from this cause alone.

The following analyses of samples of old or used charcoals are due to Wallace :—

LXXII.—ANALYSES OF VARIOUS OLD OR USED BONE CHARs.*

	D.	E.	F.	G.	H.	I.	K.	L.	M.
Carbon (nitrogenous)	9.74	10.60	12.86	19.64	7.42	10.64	5.82	17.28	2.56
Calcic phosphate	82.80	83.20	81.80	73.20	87.08	80.56	77.26	79.56	90.73
Calcic carbonate	5.92	4.15	2.92	3.18	1.92	4.52	14.66	1.05	3.50
Calcic sulphate67	.64	.42	1.12	.95	2.24	1.03	.59	1.10
Ferric oxide33	.55	.67	.66	.85	.72	.21	.69	1.17
Silicious matters54	.86	1.33	2.20	1.78	1.32	1.02	.83	.94
	100.00	100.00	100.00	100.00	100.00	100.00	100.00	100.00	100.00
Cubic feet per ton	44	39	36	32	29	35	40	34	35

* Analyses of new chars are given on p. 343.

D is first-class charcoal; E is of excellent quality; F is of fair average quality; G is pretty old and very much glazed; H is very old and over-burned; I has been used in a sugar house where hard water is employed; K has been used in a Continental beet factory; L has been soured in the process of washing; and M has been exposed to the air while cooling.

Remedying the effects of continued use and reburning on charcoal.

—In order that the charcoal may do its work effectively, the logical deduction or conclusion to be drawn from the above facts is that repeated renewal of the charcoal is very obviously necessary. It will not do to simply replace the char dust sifted out and eliminated from time to time. No more will it suffice to make good the deficiency due to continual shrinkage on reburning, by adding new charcoal. The charcoal will not do its work effectively unless an aliquot part of the whole char, and not simply the dust alone, be removed and its place filled up with quite new char. This should be done at the rate of 50 per cent. per annum, and the removal and addition made continuously—*one, two, or three bags of new charcoal to every cistern each time it is charged with reburnt charcoal.*

Effects of the quality of the water on the composition and decolorising properties of the charcoal after continued re-use and reburning.

—The small percentage of carbonate of lime present in new char is useful in neutralising the small amount of acid present in nearly all raw sugars. Moreover, there is always a certain amount of acid formed during the washing of the char by a kind of fermentation process which it is extremely difficult to prevent occurring. It is not by any means desirable in all cases to use charcoal from which the whole of the carbonate of lime has been removed. The use of such a char will certainly result in the generation of sour liquors, and give rise to iron in the syrups. Moreover, when a very soft water is

employed to dissolve the sugar, and also to wash the char, the percentage of carbonate of lime gradually diminishes, and as the char ages it may descend as low as $1\frac{1}{2}$ per cent., or in fact eventually disappear altogether. On the contrary, in the case of very hard water, the carbonate of lime either diminishes to an almost imperceptible extent, or increases often to an alarming degree. In the Continental beetroot-sugar factories, where lime is abundantly used in defecating the juice, serious inconvenience results. Many devices and schemes have been tried for the purpose of freeing the charcoal from this obnoxious ingredient when it is allowed to accumulate in the charcoal to too great an extent. It closes up the pores of the charcoal, so that it cannot fulfil its functions.

Washing with dilute hydrochloric acid.—One remedy is to wash the char with 1 or 2 per cent. of hydrochloric acid previously diluted with enough water to saturate the char. (See p. 167.)

Beanes' process.—*Treatment of the charcoal with dry hydrochloric acid gas.*—Beanes' process is based upon the fact that, whilst dry hydrochloric acid passed over dry carbonate of lime remains passive, *i.e.* it does not give rise to any chemical action whatever, yet when the same dry acid gas is passed over charcoal containing carbonate of lime the latter is completely decomposed—the more so if the charcoal be warm. The freshly burnt char is impregnated with perfectly dry hydrochloric acid gas until the charcoal is thoroughly saturated. The charcoal is then exposed to the air until the excess of gas escapes, thoroughly washed with water and again burnt.

Good results follow the treatment of the charcoal with acid in beetroot-sugar manufactories, and also in some particular instances in cane-sugar refineries, where the liquors are for some reason or another slightly alkaline. On the contrary, in those refineries where soft water is employed the treatment of the charcoal with acid is much to be deprecated, because the carbonate of lime, far from being present in excessive quantity, is not present even in sufficient amount to neutralise the very small quantity of acid present in the raw sugar.

Although it is a well-known fact that acid-treated char yields a paler filtrate than char which has not been acted on by an acid, yet it has not been found practicable to remove every trace of acid from the char by simply washing with hot water. There is therefore great liability of the sugar in subsequent operations—during the boiling-down process—being converted into fruit sugar. Moreover, the charcoal washings will be very sour, and the syrups liable to contamination with iron salts. Working in the ordinary manner, therefore, a smaller amount of syrup will be produced than if the charcoal had been treated with an acid. The sugar produced in the case of the acid-treated charcoal would, however, possess a better colour.

Advantage of applying Beanes' process to new char.—Beanes', and processes of a like nature, may be advantageously used to remove

injurious bodies in new char, so as to bring it immediately into a fit working state. Traces of ammonia and sulphide of ammonia, as well as free lime, in addition to an excess of carbonate of lime, are always present in new charcoal. Although the washing and reburning, to which all new charcoals should be subjected, remove the ammonia, carbonate of lime, and free lime, yet the excess of carbonate of lime imparts a very yellow colour to the filtrated liquors, and, as a matter of fact, generally some five or six weeks elapse before the charcoal is in prime condition. But, so long as the charcoal is added gradually in small quantities at a time, there is little or no risk of any bad result; on the contrary, an immediate decided advantage accrues from working in that way. For effect of bad washing of char, see p. 321.

SUGAR BOILING IN VACUO.

The perfectly clear and limpid syrup from the bone-char filters is then boiled in pans similar to those used in beet-sugar works (Figs. 54 and 55), but made entirely of copper. The boiling into refined sugar should yield fine regular crystals only. Boiling is done in the hot, that is, with a slight vacuum, and rapidly.

The theory of ebullition.—A liquid boils when the disruptive force of heat on the particles of the liquid is sufficient not only to dis sever them, but also to enable them to overcome the atmospheric pressure, and to cause them to be evolved in the form of vapour.

Boiling-point.—The greater the atmospheric pressure, the higher is the temperature at which the liquid boils. The lower the atmospheric pressure, the lower is the temperature at which the liquid boils. The temperature at which a liquid boils is called its "boiling-point." But the "boiling-point" of any given liquid loses much, if not all, of its significance if the pressure of the superincumbent air in which the experiment was conducted be not stated.

It will thus be seen that atmospheric pressure retards ebullition to a greater or less extent, varying with the altitude. The air is more rarefied at the summit of Mont Blanc than it is at the bottom of the valleys underneath—so much so, in fact, that, as every one knows, water boils on the top of that mountain at so low a temperature that it is impossible to cook a potato at that height. But at the present moment we are not so much concerned with the results obtained by boiling substances in water, but rather with the temperature itself at which the water boils. Moreover, the pressure of the atmosphere can be removed artificially without ascending a high mountain to a rarefied atmosphere. Again, very volatile liquids, such as ethylic ether, gasolene, etc., can be made to boil at the ordinary temperature of 60° F. by the mere removal of atmospheric pressure.

Laboratory methods of removing atmospheric pressure.—This fact can easily be demonstrated by placing a test-tube containing a little

ether under the receiver of an air-pump and exhausting the air, when the ether evolves vapour so rapidly as to enter into violent ebullition.

This method of facilitating and greatly increasing the development of vapour is in constant use in chemical laboratories. Many substances are greatly injured or altogether destroyed by expelling the water in which they are dissolved, by the application of heat in the ordinary way. But however delicate a substance may be, the water or other volatile solvent in which it is dissolved may be easily evaporated from it under the action of the air-pump. Suppose, for instance, it were required to evaporate away all the water of an aqueous sugar solution, and to obtain all the sugar in a crystalline state, the experimental chemist would accomplish this in the following manner. He would put the solution to be evaporated under the receiver of an air-pump, along with a shallow vessel containing oil of vitriol. He would now exhaust the receiver of the air-pump, the consequence of which would be an increased evolution of vapour from the sugar solution. The vapour might be removed continuously by a prolonged working of the air-pump, but the operation would be troublesome, and the sulphuric acid placed under the receiver renders the continuous working unnecessary, as it attracts and absorbs the aqueous vapour as soon as formed, thus rendering the process continuous.

The laboratory air-pump inapplicable on the large scale.—The laboratory method is, for obvious reasons, impracticable on the large scale. If applied at all, it would have to be greatly altered. This was accomplished, in the first instance, in a very ingenious and satisfactory manner by means of the vacuum pan (Fig. 78) invented by Howard, and which is lasting evidence of his constructive skill.

Description of Howard's vacuum pan.—The vacuum pan consisted in the beginning of two shells of copper of spherical or spheroidal curve, s and s' ; the edges of both were flanged, and the flanges screwed together.

The steam jacket.—The lower of these copper shells was surrounded externally by a steam jacket f , by means of which the contents of the vacuum pan may be heated.

The steam coil.—A spiral copper worm, ramifying internally, serves as a supplementary source of heat. Through the middle part of the lower copper shell, and through the steam jacket which immediately surrounds it, is an aperture P , capable of being shut or opened at pleasure, externally, by means of a valve with ground edges. This aperture is for the purpose of allowing the contents of the vacuum pan, when sufficiently boiled, to escape. Directing our attention to the apertures in the upper copper shell, we find them more numerous.

Manhole.—First there is a *manhole* m , exactly similar to the manhole in the boiler of a steam engine, through which a workman

enters for the purpose of cleaning the interior, effecting repairs, etc. This large aperture, like the corresponding one immediately opposite in the lower section, is capable of being accurately closed by means of a valve with ground edges.

Barometers, thermometers, and feeding apertures.—The next apertures to be described in the upper section of a vacuum pan are those for admitting a *barometer* and a *thermometer* respectively, *b* and *t*, each being inserted through its own air-tight packing. Then comes an aperture through which the sugar solution is introduced.

How the contents of the pan are sampled without introduction of air.—*The proof-stick and its functions.*—Lastly comes an arrangement for the very ingenious purpose of allowing a portion of the

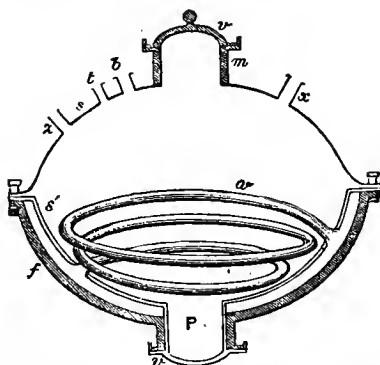


FIG. 78.—Diagrammatic representation of Howard's vacuum pan (cp. Figs. 54 and 55).

contents of the pan to be withdrawn without letting in air, and consequently destroying the vacuum. It is exceedingly difficult, without actual examination, to give a clear notion of the method by which this problem is solved. A general description, however, is as follows. Into an aperture leading into the interior of the pan one end of a brass pipe is securely fastened (air-tight, of course), and projecting its other end to within 6 inches of the lowest part of the vacuum pan. This lower end, which when the pan is charged is necessarily covered by the charge, is furnished with a rotary spill like the spill of a cask, and which may be turned off or on at pleasure by means of a key termed the "proof-stick," which projects to the bottom of the sheath. When turned on, the little excavation *i* (Fig. 79, 2) is made to coincide with the lateral aperture *m* (Fig. 79, 1) of the sheath, and the pan is said to be open. We will suppose, however, the operation of taking a proof to commence when the pan is *shut*,—in other words, when the excavation in the spindle does not coincide

with the lateral orifice in the sheath. Whilst the arrangement remains as described, the long key or proof-stick is thrust down, its lateral aperture (*i*, Fig. 79, 2) being made to correspond with the lateral aperture of the spindle represented in Fig. 79, 2. The proof-stick being now turned through an arc of two quadrants, both its own lateral aperture and that of the spindle revolving with it are brought into coincidence with the lateral aperture (*m*) of the sheath; and the sugar solution flows into the cup-like depression, or lateral excavation of the proof-stick. Supposing the latter to be now removed *without turning*, the vacuum would be destroyed; but if it be turned back into its original position, through an arc of two quadrants, so as to be no longer in coincidence with the lateral aperture of the sheath, and then removed, a portion of sugar solution will be brought away without in any way affecting the vacuum.

The wet air-pump; its function and mode of action.—The liquor is made to flow into the pan by taking advantage of the suction power of the instrument attached to the vacuum pan for the purpose

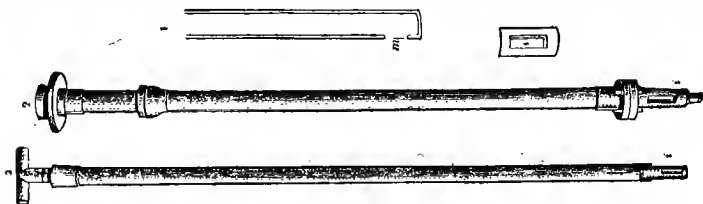


FIG. 79.—Proof-stick. 1, sheath; 2, proof-stick and its spindle; *i*, lateral aperture of proof-stick; *m*, lateral aperture of sheath.

of creating exhaustion, and termed an air-pump (see Figs. 46A, 49, and 50, and context), although it is not an air-pump in the sense of the instrument commonly bearing that name. It pumps more water than air, and without water it will not act. It may be readily imagined that if a very large air-pump, such as we use in laboratories, only much exceeding the latter in size, were attached to a vacuum pan already containing liquid sugar solution, and that liquid heated by means of the steam jacket externally applied, and of the worm coil internally, the tendency towards a vacuum would be induced, and ebullition at a reduced temperature would ensue. In practice, however, the use of such an air-pump as this would be altogether out of the question. In the first place, its size as compared with the ordinary vacuum pan would be enormous. In the second place, the delicacy of its valves would be such as to render them soon worthless, hence some modifications of the common air-pump is desirable. If we cannot employ an air, or rather a steam, pump, the first suggestion that occurs is, whether the steam might not be condensed into water, and the water pumped

away instead of the steam itself? This suggestion has been fully carried out in what is called the *air-pump* of a vacuum pan, and which is in every respect similar to the air-pump of a low-pressure steam engine. It is an instrument of the simplest possible construction, merely consisting of an iron cylinder, into which fits a piston, supplied with one or more metal door-like valves, opening upwards. The action of such a pump as this will be evident from a mere examination of its parts. The piston being pressed down, the door-valve *will have a tendency to rise*, and, being raised up, the door valve *will have a tendency to fall*. Now, whether the tendency be brought into active operation or not will depend altogether on the medium with which the pump cylinder may be filled. Any one conversant with the nature of pumps will at once see that such an instrument as this could not pump air effectually, inasmuch as large volumes would pass between the valve and its vent. No less obvious is it that such a pump would be very efficient, provided the object were to pump water.

Condensers.—It follows, therefore, from a consideration of these peculiarities, that the vapour evolved from the solution under ebullition must pass into an intermediate vessel and become condensed before the so-called air-pump could act. Accordingly, the latter, instead of being directly connected to the vacuum pan itself, is joined to an instrument called the condenser (Figs. 46A, 49, and 50).

Injection condenser.—Condensers assume various modifications both in low-pressure steam engines and vacuum pans. They resolve themselves, however, into two classes, each having its own distinctive type. Either condensation is effected by the injection of water amidst the steam, or the cold water is made to circulate through a series of tubes with which the steam comes in contact externally. Condensers of the former type are the most simple and the most general.

Description.—An injection condenser is a very simple instrument, merely consisting of a large cylinder, having within it a tube or tubes communicating with a tank of water externally, and perforated. The consequence of this arrangement is that, as soon as the air-pump is set in action, jets of water escape from these apertures and, rushing amidst the steam, convert it into water now easily recoverable by the pump.

It therefore follows, from the combined agency of all these appliances, that the refiner brings his dense sugar solution to a violent ebullition at a temperature rarely exceeding 150° F.,—indeed much lower if required,—and thus avoids much of the injurious consequences resulting from the application of heat. Nevertheless, a considerable amount of destruction is effected, even by the vacuum pan, as may be recognised in the final result; for though the liquor originally exposed to evaporation may be, and frequently is, absolutely without colour, the result, after boiling, is more or less yellow, and the amount of tint is always proportionate.

PRACTICAL INSTRUCTIONS FOR USING THE VACUUM PAN

History.—According to Dr. Scoffern, ever since the discovery of the vacuum pan in the early part of the nineteenth century, the method of using it was veiled in secrecy. He had had communication at different periods up to 1853 with the greater number of metropolitan, several country refiners, and with a still greater number abroad. Of British refiners, he tells us he had only met with one principal who had a sufficient knowledge of the vacuum pan to conduct an operation of boiling. By far the greater number were so unacquainted with the machine that they could not check the work of the boiler by reference to theoretical knowledge of the philosophic principles on which the action of the vacuum pan depends. These remarks did not apply to Continental refiners, most of whom even then possessed not merely a theoretical but a practical acquaintance with the vacuum pan, and could use it in the absence of a boiler.

It is strange, comments Dr. Scoffern, that the community of “boilers”—most of them Germans—who were engaged in English refineries managed so long to turn their secret to profitable account. They succeeded in creating the impression amongst their subordinates that any attempt to work the vacuum pan without previously making an honorarium of at least ten guineas to the boiler would be futile.

Ten guineas was the *usual sum* paid by a pan-man, who is a sort of sugar boiler's assistant, before he was allowed to take a proof and shown how to put in the grain, this being the technical expression amongst sugar boilers for the generation of crystals. Nevertheless, it happens that the pan-man may possibly have conducted thousands of boilings, each through nine-tenths of its total duration, for the boiler usually devotes his attention to no more than two phases of the evaporative operation; he stands by, taking proofs until the first crystals have appeared (putting in the grain), and, being called by his subordinate when the pan has got its full charge, he determines when the proper degree of evaporation has been arrived at, consequently when the steam should be cut off and the evaporative liquor allowed to escape from the vacuum pan by opening the valve.

Occasionally—but far more rarely than prudence should dictate—a stranger, perhaps a colonist, presented himself at a refinery for the purpose of learning the secret. Then the prescriptive fee of ten guineas was departed from, and the student was charged a sum ridiculously disproportioned to the amount of information conveyed.

Nevertheless, the vacuum pan is an instrument which depends on a combination of chemical and mechanical principles. Its particular use, too, cannot be learned by mere directions; nevertheless, directions can be so given that any person of ordinary capacity by following them may become, after a few operations, a good practical boiler.

In the first place, then, the student should consider the nature

and objects of the vacuum pan (Fig. 78). It is a copper shell composed of two hemispheroids—the lower one embedded in a steam jacket, the upper one not so embedded. In addition to the steam jacket *f*, external to the lower hemispheroid, there is, internal to the same, and coiling spirally within, a copper steam tube *a*, technically called a worm.

In the lower hemispheroid of the vacuum pan there is only one opening P. This is for the purpose of letting off the liquor when sufficiently evaporated. It is closed by means of a valve or door, opening downwards, and acted upon by a lever (Fig. 83, D, C). Its edges are ground perfectly air-tight against a rim, and the door should be capable of closing accurately by means of oiled contact alone. Nevertheless, in practice, and with pans that have been some time in use, this accurate contact is not to be depended upon; hence it is a safe plan, and one usually followed, to stop all chance of air leakage by pouring a little syrup into a circular depression, which surrounds the ground edge of the door valve, and supplied with this intention. A similar remark is applicable to the manhole cover *v* and its corresponding rim, to be described hereafter in connection with the upper hemispheroid. These large ground surfaces, in consequence of their great extent, scarcely admit of any other treatment in practice, but it should not by any means be extended to the small ground joints (Fig. 78, *b*, *t*) corresponding with the barometer and thermometer. There can be no excuse for employing, for the purpose of rendering them air-tight, any other liquid than oil sparingly applied.

The testing of a vacuum pan.—Applying steam.—It is assumed the operator is about to try the efficiency of a new vacuum pan never yet used. We shall therefore trace the operation of boiling in it a charge of sugar from beginning to end. The first points to be ascertained are the condition of the air-pump and the state of the pan as regards tightness. The operator commences first by setting in action the air-pump; he then turns on a small amount of water into the condenser for the purpose of moistening the valves of the pump, without which care the result of pumping will be inefficient, the so-called air-pump not being intended to pump air, but a mixture of air and water. These directions having been attended to, the barometer gauge is to be carefully regarded. If the vacuum could be rendered perfect (which it cannot), the mercury would stand at the same level in both legs of the barometer gauge. In proportion as such an equality of level is reached or departed from, so will the instrument be in good or bad order. If the mercury refuse to stand, but rise and fall fitfully with every beat and counter-beat of the piston, then there is a leak somewhere. When such a leak depends on a natural imperfection of the vacuum pan, and not on a casualty such as the interposition of a grain of sand or other foreign body in either of the ground joints, then its discovery belongs rather to the

engineer than to the sugar manufacturer; nevertheless, for the guidance of colonists and others who may chance to have a vacuum pan and no engineer, a few remarks may properly be given. To determine the exact ground surface to which the leakage is due, it will generally suffice to apply the ear to each in succession, when generally the hissing sound occasioned by the rushing in of atmospheric air will be sufficiently indicative of the leakage. Occasionally other plans must be had recourse to, the most generally eligible amongst which consists in pouring oil or water externally on the ground joint, and noticing whether it remains there or whether it be forced inward by reason of atmospheric pressure. Should both these means fail to indicate the locality of the mischief, then each joint must be examined in the following manner; indeed, the operation now to be described has to be performed even though the leak have been already referred to one specific ground surface. In the latter case, for the purpose of showing the exact point of imperfection on that surface, we will suppose the ground bearings of the manhole to be examined; for which purpose the ground surfaces are to be well oiled, then the oil wiped off, and the surfaces finally dried by means of a piece of rag or a little cotton waste. A thin paste is now to be made of vermilion and oil, or red-lead and oil, a portion of either of which is to be smeared either on the ground surface of the cover or on the ground surface of the bearing, but not on both. The cover is now to be placed *in situ*, and revolved under pressure in such a manner that every portion of its ground surface may be brought into contact successively with every part of the bearing. The rationale of the treatment with red pigment will now be obvious. If any inequality of bearing exist, such will be evidenced by interrupted spaces, over portions of which the red pigment has become attached, whilst other portions remain uncovered. It is evident that, if the ground surface had been perfectly accurate, the imprint of red pigment would have been equally impressed. It is evident, moreover, that the smeared portions of the ground surface will be those requiring to be lowered in order that the joint shall be rendered accurate. This can be, and indeed is, accomplished by careful filing and scraping. A far shorter process, however, suffices for those small tap-like joints which belong to the barometer gauge and the thermometer (Fig. 78, *b*, *t*); should either of these joints be imperfect, they may be rendered accurate by means of grinding, a little fine emery and oil being used for that purpose. The operation is conducted by smearing the tap-like portion of the ground joint with the oil and emery, then rotating the latter in its sheath until the joint has been rendered perfect, which may be ascertained by means of the red pigment. We shall now suppose the pan to be proved to be in good order, so far as relates to external appearance; the next point to be investigated is whether it be tight as regards steam. The act of turning on steam is one which

demands a few remarks relative to precautions necessary to be taken in order to guard against rupture or explosion. In the first place, it may be laid down as a general rule that a steam tap, with whatever vessel communicating, should never be turned suddenly and at once, but gradually. Neglect of this precaution often leads to dangerous accidents, and always exposes the pipes, joints, and vessels immediately concerned to risk of fracture. There is another precaution to be observed in the operation of letting steam into the jacket or worm of a vacuum pan, or indeed into any closed vessel. Simultaneously with the entrance of the steam, the air contained in such vessel must be allowed to escape through a tap supplied with this object. The rationale of this operation will be evident on slight consideration. It is this: the steam (always under pressure) enters the closed air vessel, and, disposing of a portion of its latent heat without itself getting less hot, heats, and consequently expands, the air contained in the vessel. Hence the latter has to retain the pressure, not of steam alone but of a mixture of steam and air. Consequently, before turning steam on to the vacuum pan, or any vessel constructed on a similar principle, it is always necessary to previously open an air-tap, for the purpose of allowing air to escape. The tap should be retained open until steam is observed to issue violently from it: it should then be closed. The practice of retaining it a little open during the whole operation of boiling, as is followed by some sugar boilers, is totally unnecessary.

Pressure of steam.—It now remains to treat of the pressure of steam best adapted for working the vacuum pan. In the greater number of sugar houses this pressure is not allowed to exceed 10 lbs. to the inch; it may be that a still lower pressure, say 7 or 8 lbs. to the inch, is even better. At any rate, steam of above 10 lbs. to the inch should not be employed: firstly, because its heating power (under the circumstances of its employment) is not so great as that of steam at a lower pressure; secondly, because of the dangerous strain to which high-pressure steam subjects the apparatus.

In stating that high-pressure steam has not so great a heating power as low-pressure steam, the remark must be limited to the condition of heating a fluid by means of a steam-warmed jacket or coil. If a fluid be heated by injecting steam into and through it, then high-pressure steam, weight for weight, is the more powerful.

Inasmuch as the greater number of refineries possess high-pressure engines, although low-pressure is necessary for working the vacuum pan, an intermediate receptacle called the expansion vessel is required. It consists of a metallic cylinder, to which a valve is attached, weighted to the degree of maximum pressure which the contained steam is allowed to reach, and which pressure once exceeded, the valve opens and, facilitating the escape of a portion of the steam, maintains the desired equalisation.

Condense box.—Proceeding with our investigation of the successive phases of action which develop themselves on admitting steam into the jacket and copper worm of a vacuum pan, we shall presently find the necessity of an adjunct as yet undescribed. The steam portions and appendages of the vacuum pan being only heated to the temperature of the surrounding atmosphere anterior to the admission of steam, it follows that a certain amount of steam will be condensed into water by the operation of cooling. It is certain, moreover, that such cooling agency will be always in operation; therefore, except some provision were made for removing the water thus resulting from condensation of steam, the jacket and worm would soon be rendered inoperative. Such provision is made by an instrument termed the *condense box*. To describe the mechanism of this apparatus is unnecessary, seeing that, its principle being enunciated, a slight examination of the mechanism of the apparatus will show in what manner the conditions are applied to practice. The “condense box” must necessarily be fixed on a lower level than that of the vacuum pan, from which it is usually some considerable distance removed.

Testing steam jacket and steam coil for steam-worthiness.—Let us now return to an examination of the pan itself. We have already determined that those portions of it not having immediate reference to steam are tight and trustworthy; it remains, therefore, to examine the condition of the steam jacket and coil. For this purpose, the steam being laid on with all the precautions already detailed, the operator places a strong light near the lens orifice and looks into the pan through the sight-hole opposite the former; or, turning off the flow of water into the condenser (not to be confounded with the condense box), stopping the air-pump and removing the manhole door, the observer may stand on the rim of the pan and look through the manhole. It is possible to conceive a case of steam leakage under the aid of a vacuum ceasing to be a leakage when the vacuum is removed; such a case, however, is so exceedingly improbable that the examination of a vacuum pan as to steam-worthiness through the manhole may be regarded as perfectly reliable; indeed, on account of the larger and more ready sphere of vision thus commanded, the plan is preferable to an examination of a pan through the sight-hole.

Any defect as regards steam-worthiness can easily be observed, and its locality no less easily determined, by noticing the exact point at which the steam escapes. The escape may be either internal or external. It may be from a crack in the substance of the steam jacket or from a defective joint. If the former, the defect is fatal; the jacket must be replaced by another. If on a joint, it may be remedied by unscrewing the latter and replacing it with tow and a mixture of red and white lead; if in the worm, the latter must be removed and the defective part made good by brazing. Wherever a steam

leak exists, and from whatever cause, it is always to be regarded as a serious impediment; but next to a crack in the jacket, the existence of which renders an explosion imminent, a defective worm is the most grave condition. The consequence of the latter is that a charge of sugar under process of boiling continually undergoes a joint operation of dilution and evaporation, thus *pro tanto* destroying the specific advantages of the vacuum pan.

The actual process of boiling in vacuo.—Let us now assume the vacuum pan to have been determined perfectly efficient in every respect, and the process of boiling to be commenced. The operator begins by first setting in motion the air-pump, then turning on the condensing jets, opening the air-tap in the vacuum pan jacket, and gradually turning on the steam. He now begins to charge the pan, keeping correct account, measure by measure, of all the liquor which goes in. Of measures there are two kinds—one kind open, the other closed. The latter is most frequently employed, possessing the advantage of drawing its supply of liquor from a lower floor than that on which the pan is placed, such being in most refineries the place of greatest eligibility for the liquor cistern. It need scarcely be indicated that closed measures, as they are called, communicate by means of a tube with the liquor cistern, through which the liquor ascends into the measure, by the force of the suction exercised by the pan itself.

The total number of measures of liquor which a vacuum pan is capable of working being known, about one-third of that total quantity should be allowed to enter, and should be submitted to the full force of the pan—that is to say, the act of ebullition may be carried to any extent short of that which would result in boiling over. At this stage no harm can accrue from carrying on evaporation with rapidity, and besides other advantages a considerable saving in time will be gained. It is not to *weak non-concentrated* liquors that even the greatest evaporative power commanded by the vacuum pan can be injurious, but to the *concentrated result* of evaporation hereafter to be described.

The condensers: their capacity and rational working.—If the liquor be of good quality the boiler has very little to be solicitous about at this stage of the operation, further than observing that every function of the pan is being well performed. He need take no account of the liquor within. There is, however, one function of great importance in itself, and the consideration of which boilers too frequently neglect,—and that is *condensation*. If this be not adequately performed, much of the efficiency of the vacuum pan is lost. In order that condensation may exercise its maximum effect without overbalancing the exhaustive action of the pump on the one hand, or permitting a portion of steam to remain uncondensed on the other, some care is necessary. The condenser should never feel hot. There-

fore, if such elevation of temperature be recognised on applying the hand externally, more water should be turned on. If, notwithstanding the supply of the maximum amount of water, compatible with circumstances presently to be described, the condenser is still hot, the steam power of the pan must be diminished by a special turning of the tap. The circumstances acting as a limit to the quantity of water capable of being brought into action by any given condenser are chiefly—(1) Size of the condenser; (2) number and size of jets communicating with it; (3) power of the air-pump. If, from any combination of circumstances, the condenser be overloaded with water, the latter, instead of being removed altogether by the pump, flows back into the vacuum pan, and, mingling with the sugar solution, proves very detrimental to the ultimate result.

From a consideration of the principles on which the condenser of a vacuum pan is based, it follows that in proportion as the fluid to be evaporated is less dense or more watery, so will the amount to be removed by evaporation be greater; hence the size and power of the condenser must have immediate reference to the purpose for which the pan is intended. A power of condensation amply sufficient for the necessities of a vacuum pan, when employed in concentrating the dense liquors of a refinery, may prove totally inadequate to the altered necessities of colonial manufacture, where the juice to be evaporated is more watery. Scoffern relates on one occasion he conducted the evaporation of cane juice to crystallised sugar altogether by means of a vacuum pan. The condenser was one of great power, and the command of water unlimited; had it not been for these circumstances, the operation could not have been carried on to a successful termination.

Returning now to the liquor under operation in the pan, it will be time to examine, by taking a proof, to what extent the evaporation has been pushed. The original quantity of liquor in the pan is supposed to have been a third of the pan's total charge, its density is supposed to have been 28° B. (sp. gr. 1.245), its quality the best. Under these circumstances, supposing the evaporative operation to have been briskly carried on for about half an hour, crystals should begin to manifest themselves. The only certain way of recognising their presence, judging of their size and their general qualities, consists in taking a proof, full directions for accomplishing which will presently be given.

Decrepitation in pan as crystals begin to form.—There are other indications, however, of the formation of crystals which the operator should learn to appreciate. One of these is the peculiar crackling sound, like that of salt thrown on the fire, recognisable, as soon as crystals have formed, on applying the ear to the side of the vacuum pan. It appears to depend on an alteration in boiling of the solution of sugar in which crystals have begun to form.

Previous to this period it is viscous, *tenacious*; subsequently to the formation of the crystals the physical character of the liquid alters. It is more thin, less tenacious, and the angular crystalline particles which it contains exercise a physical effect on the operation of boiling, which now takes place with greater rapidity than before, and with a series of sharp but minute explosions. This peculiar phenomenon, which may be called decrepitation, is especially noticeable where a solution of pure cane sugar is operated upon. The hoiler should lose no opportunity of making himself familiar with so important an indication.

The flash.—Another sign of the formation of crystals in the liquid undergoing evaporation consists in what is technically called “the flash.” This is a sort of sparkling, recognisable on looking down upon the charge through the sight-hole. The flash is due to the operation of certain reflective and refractive effects of the crystals on the beam of light shining down upon them from the lamp.

Of taking the proof.—This is the great secret of vacuum boiling, making known to the operator the exact condition of things inside. It is accomplished by means of the very ingenious contrivance termed the “proof-stick.”

It will facilitate the comprehension of this part of the subject if we treat separately of the method of getting out a proof, and the examination of this proof when got out. The operation of getting out a proof requires four distinct manipulative acts, all of which it is necessary for the practical sugar boiler to comprehend thoroughly. They may be described as—

- (1) Insertion, or position 1.
- (2) Half-turn upwards to the left, or position 2.
- (3) Half-turn downwards, ending at position 1.
- (4) Withdrawal, position 1 being maintained for about the first six inches, then position 2.

Consequently it appears that, the proof-stick having been properly inserted, the remaining portion of the manipulations is a matter of simplicity. Now, as regards this insertion, it must always be remembered to thrust down the proof-stick with the scooped portion of its extremity (Fig. 79, *i*) looking downwards. Of course there can arise no difficulty in respect of this, but frequently it happens that after the proof-stick has been allowed to remain some time in the sheath the operator forgets in which direction this scooped portion of the extremity lies, whether upwards or downwards, hence an additional means of discrimination is necessary. This is provided by the maker in the following manner. The handle of every proof-stick is marked with a little round excavation on the same side as the scoop at the extremity, whence it follows that, whether the proof-stick be within or without the sheath, the exact position of its scooped extremity can be readily determined.

The examination of the proof.—Returning now to the operation of examining the proof withdrawn. Next to the operator is placed a light, a gas light on account of its brilliancy being preferable. Usually this light is placed in a box the sides of which are painted dark, though this arrangement is not indispensable. The object of the light is to enable the operator to examine the condition of the proof by the transmission through it of luminous rays, by which means, if any crystals are present, they may be readily seen long before they are large enough to be felt. The means adopted for transmitting luminous rays through the evaporated material consist in throwing it out from the scooped end of the proof-stick upon the thumb of the left hand, approaching the fore finger, separating it, and thus extending the material to be examined in the form of a sheet, which being held between the source of light and the eye, the presence or absence of crystals is readily determined. Supposing the operator to be satisfied that crystals, technically called grains, really exist, he has nothing more to concern himself about just at present, further than to take care that the evaporative operation go steadily on; under which treatment the crystals will soon be found to increase in size and in number; to become tangible as well as visible, feeling between the fingers like particles of sand, which in proportion as they are large and hard, in such proportion are they better—at least, according to English commercial notions. On the Continent, however, the production of small-grained sugar is aimed at, and indeed whiteness, *cæteris paribus*, is in direct proportion to smallness of crystal.

At this stage of the operation the practice of boiling differs in the hands of various operators—as indeed it must, according to the quality of material exposed to the evaporative action, and the exact condition of staple required. As a general rule, however, it is proper to carry the evaporative process to such a pitch that a proof can barely be attained on account of the thickness of material in the pan. When this state is arrived at, then it is time to increase the amount of material by turning on a flow of liquor from the measure into the pan.

Feeding the pan: (a) by gushes, (b) by gradual infusion.—There are two distinct methods of feeding the pan,—either by gushes or by a process of gradual infusion. The latter is in theory the most scientific; it is that most generally adopted by intelligent sugar boilers, and is in every way the best method. In practice it is no less easy than it is comprehensible in theory, and consists in so arranging the flow of liquid that the amount of liquid allowed to enter the pan may be proportionate to the force of evaporation. If it be less, then the contents of the pan will become too thick; if it be more, then, supposing the operation of evaporation to have been continued sufficiently long, it is evident that the crystals already formed will become dissolved. This latter is one of the most unfavourable state of things which can occur, but it may always be

prevented by attention on the part of the boiler, who, by means of frequently taking proofs, can always make himself acquainted with the exact condition of his charge (cp. Fig. 61, p. 278, and context).

Final precautions.—When the last portion of liquor is already in the measure, and consequently the operation of boiling almost finished, the boiler must pay great attention to his charge. The quality of his resulting sugar loaves will in great measure depend upon his causing the operation of boiling to cease at exactly the proper instant of time, a few seconds more or less making great practical variations in the results. Nothing but practice—very extended practice combined with great intelligence—can supply the needful knowledge; by due attention to theory and general principles, the errors of boiling may be restricted within the range of comparatively narrow limits. Perhaps the following general directions for concluding the operation of boiling are the best, though considerable manipulative discrepancies exist in the practice of different operators. Whether the system of feeding the vacuum pan by sudden gushes or gradual infusion has been adopted, it is always a safe plan for the boiler to adopt the former, as concerns the last measure of liquor. With whatever degree of care the turn of the liquor-tap may have been adjusted, it will be generally impossible to arrange it precisely at that point conducive to the best final result. For instance, at the point when all the liquor save the last measureful has been turned into the pan, and a proof being taken, the operator probably will find that the result is either a little too thin (free) or a little too thick (stiff). Such may happen to charges under the care of even the most experienced boilers. Where the system of gradual infusion is allowed to proceed, this undue stiffness or thinness would continue to the end of the operation; whereas, by taking the last measureful of liquor under his immediate control, the boiler has it in his power to modify this state of things, and bring the evaporative process to the condition finally required. With regard to the exact condition of stiffness at which the operation should be concluded, and the contents of the pan turned off into the heater (Fig. 81, G), there is a considerable difference of practice. Supposing the material to be of the best quality, the operator may be told as a general rule to boil up to the degree of thickness at which a proof can barely be taken. Admirable results have been known to be produced from the contents of a vacuum pan boiled so very stiffly that not only could a proof no longer be taken, but the material was obliged to be forced out of the pan by a pole inserted through the manhole. This material, however, was the result of liquor which had been purified by means of lead (p. 344). Sugars defecated by lime and bullock's blood in the old-fashioned manner contain such an amount of glutinous adhesive matter that an evaporation carried to the extent described would have been totally incompatible with the subsequent operations of claying, liquoring, and

drainage, operations on the successful performance of which the perfection of the final product depends.

Immediately the evaporative operation has been carried to the furthest extent desired by the boiler, the process should be stopped. First of all, the steam communication is cut off; then the flow of water into the condenser; next, the air-pump; next, an air-plug being removed, or a tap being opened (contrivances vary in different pans), air is admitted into the pan; and finally, the let-off valves C or C' being opened by the lever D or D' (Fig. 81), the charge escapes into the heater G below. At this period a tap is turned, by means of which a jet of steam is injected into the pan, for the purpose of freeing it from adherent sugar.

Boiling by doubles.—Occasionally, when the liquor to be evaporated is of inferior quality, the ordinary conditions of boiling already described are not capable of yielding a sufficient crop of well-defined crystals. Under these circumstances the operation of boiling by doubles, as it is technically called, is frequently had recourse to. The theory on which it is based rests on the fact that crystalline nuclei being placed under favourable circumstances in a vehicle containing similar material in solution, crystalline molecules are not only deposited on the nuclei already existing, but the formation of additional crystals is determined. In the operation of vacuum-pan boiling this principle is readily applied by retaining a small portion of one charge as a foundation for the next (see pp. 277-279).

Boiling by the thread proof.—The operation of vacuum-pan boiling, as already described, has been conducted on the supposition that crystals are formed during the evaporative operation, and the gradual progress of such boiling has been judged of in reference to the appearance of crystals formed as indicated by the proof. Such a result, however, only applies to liquors which are moderately good. In proportion as the material to be boiled becomes laden with glucose and other impurities, so will its tendency to generate crystals during the evaporative act diminish; and, in the course of the actual routine of a refinery, many charges will have to be submitted to the action of the pan which no manipulative skill could succeed in granulating under the operation of boiling; hence the method of judging by proof requires to be varied, seeing that no crystals exist, for the guidance of the boiler. Recourse must then be had to the indication afforded by the length, brittleness, and some other characteristics, of a string of the proof, formed by bringing a portion of the evaporated mass between the finger and thumb, and separating them. Three distinct phases of manifestation will be rendered evident under this treatment. The string may be formed of the length of two inches or more. After existing for the period of a second or two the string breaks midway, each end rapidly, almost suddenly, shortens, returns respectively on the finger and thumb, and be-

comes lost in the two portions of the original mass. This indication serves to show that the material has not been boiled enough for any final purpose. Supposing the evaporative operation to be still pursued, the thread, after breaking as before, forms a hook at either extremity, which is indicative of the fact that the evaporative process has been almost carried far enough. The evaporation being still further continued, the string breaks as before, but its extremities form little spirals, each something like the spiral of a corkscrew, the thread still retaining its power, though in a diminished degree, of collapsing on the finger and thumb. Beyond this degree of evaporation the operation should not be carried; if pushed to such an extent that the collapsibility of the thread is lost, the final result will drain with difficulty or not at all,—in other words, the operation will have been a failure.

Cleansing the proof-stick.—This point in vacuum-pan boiling is one of such great importance, and the neglect of it will so completely destroy the efficiency of the proof apparatus, that for all practical purposes the latter might as well not have existed. Nothing but the most scrupulous attention to cleanliness will maintain it in good working condition, to say nothing of the chances of accident to the apparatus from attempts to wrench out the proof-stick when it has become firmly set in its sheath by means of consolidated sugar.

The cleansing of the proof apparatus involves two operations—(1) The cleansing of the proof-stick, and (2) the cleansing of its sheath. We shall now give a description of both operations, commencing with the former. The proof-stick having been removed and the proof examined, the operator, by means of a bit of chip, clears out the proof hollow; he then plunges the instrument into a pail of water, withdraws it, and wipes it dry. Finally, he oils slightly the two ground or bearing portions of the instrument, namely, the ground ring immediately contiguous to the handle, and the extreme further end. These operations being performed, the proof-stick may be regarded as cleansed, and ready to be employed in taking another proof. The operation of cleansing the proof-sheath is accomplished by sponging—that is to say, a little piece of sponge or cotton waste or soft rag, being firmly attached to the extremity of a rod, is moistened with water, plunged down into the extremity of the sheath, rotated several turns, finally withdrawn, washed, and the operation repeated as often as may be necessary, until the cleansing has been accomplished, when, finally, the external ground socket of the proof-sheath is slightly moistened with oil. In conducting the operation of cleansing the proof-sheath, two special points present themselves for remembrance. The rag, cotton waste, sponge, or other cleansing material, attached to the extremity of the rod already described, must be very firmly attached, otherwise there will be great danger of its remaining in the proof-sheath. Should this ever occur the soft material must be removed at once, which can be best effected by the screw end of a

gun ramrod. The second especial point for remembrance is this: the sheath, although it must be perfectly clean, should never be allowed to retain any considerable portion of the water wherewith it may have been washed—that is to say, although it is a good plan to begin operations by thoroughly saturating the sponge of the cleansing stick with water, yet this water, having served its purpose, should be finally removed by insertion, withdrawal, and squeezing of the sponge. The propriety of this care will be evident on considering that the operator judges of the progress of his boiling process mainly by the consistency of his proof. In proportion as the latter is thick or thin, so is it inferred that the evaporative process has advanced or been retarded; but if the water due to the cleansing sponge be not accurately withdrawn from the proof-sheath, it is evident that a fallacious indication of thinness will be manifested.

Reheaters.—When the vessel is full, and when it has been found after many trials that the boiling is finished, a valve situated in the lower hemisphere of the pan is opened and its contents run into a capacious copper vessel (Fig. 81) placed immediately below the vacuum pan, usually on another floor provided with a steam jacket, and furnished with a large tap or a discharge sluice. This vessel is constructed, in fact, like the lower half of the vacuum pan, *i.e.* it consists of a copper shell enveloped in a steam jacket. It has not, however, like the lower segment of a vacuum pan, a copper coil or worm. The boiling is kept up to about 80° C. (176° F.) the whole of the time it is in the reheaters. Heat is kept up exclusively by the jacket. The vessel which we have just described is termed by some refiners the “heater,” by others the “cooler.” The reason for this apparent anomaly is to be found in the fact that, whatever be the temperature at which the solution of sugar has been boiled, it requires to be “potted” at a temperature lying between 170° and 185° F. But when the old method of boiling, namely the open-pan method, was in vogue, it necessitated a final temperature of 230° F., and often even a still higher one. In that case it followed, as a natural sequence, that the vessel into which the boiled mass was poured before potting was in reality a cooler. But since the vacuum pan came into use in sugar refineries for boiling down sugars, the temperature at which the operation is conducted being in most instances lower than 160° F., it therefore follows that the vessel in which this boiled mass is raised to a temperature of 170° or 180° F. acts as a heater. This is the cause of the anomaly in the diametrically opposite terms used to designate this vessel.

The rational use of this heater requires much skill and experience. Its scientific use depends on the physical law that there is a certain heat within the comparatively narrow limits of which those solutions which are capable of being crystallised have a maximum tendency to assume the crystalline condition. As it comes from the vacuum pan

the boiled mass should have assumed a decidedly crystalline condition during boiling—that is, unless the charge be one of a “low” nature

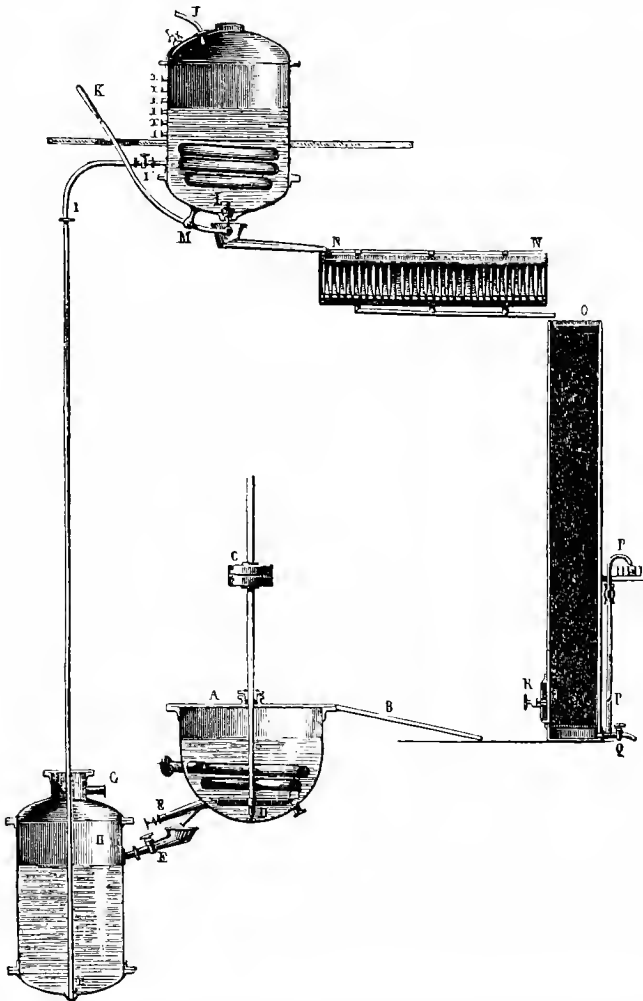


FIG. 80.—Diagrammatic representation of sugar refining.—A, blow-up pan; H I, montejus; L, defecation pan; K M, discharge lever and valve; N N, Taylor's filters; O, bone-char filter; P P, swan-neck for feeding pipes conveying filtered sugar liquor to vacuum pan.

containing large proportions of glucose and colouring matters, and which can only be wrought up into the brown sugars known as "pieces" and "bastards." In any case, the object of the heater is to still further increase the conditions favourable to crystallisation. This is done by raising the temperature up to a certain limit, according to the nature of the charge, good sugar being potted at a lower temperature than poor qualities. In the case of first-class loaves the temperature of 80° (176° F.) is the best.

The extra heat required to raise the temperature is got by applying steam to the jacket, and during the time the heat is getting up the mass is kept agitated.

It has been observed that the functions of the heater and its

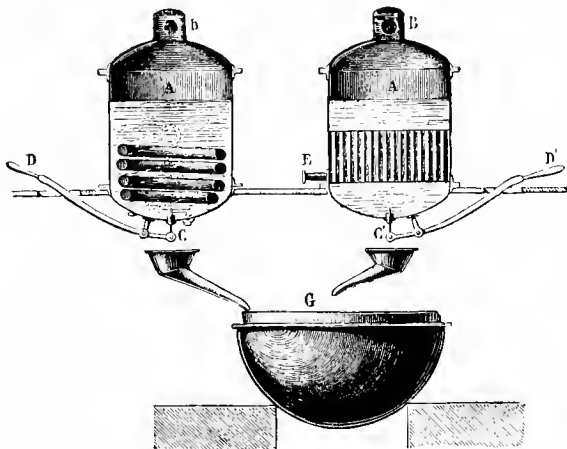


FIG. 81.—A A, vacuum pans ; C C', let-off valve ; D D', discharge lever ; G, reheater.

rational working have not received the attention they deserved. The general routine of sugar-house custom is to heat all the boiled masses, whatever their nature and their origin, to the same temperature in the heater, without regard to quality or consistency. This is a bad practice. By modifying the temperature to which the boiled mass is brought in the heater, many defects of boiling, such as too great freeness or stiffness, may be obviated. One of the many conditions favourable to the perfect growth and development of crystals is perfect freedom and liberty of motion between the particles or molecules about to assume the crystalline condition. But, as a matter of fact, the temperature most favourable to the crystallisation of the sugar particles or molecules is considerably higher than that which can be maintained during the whole of the time occupied in boiling without

destroying a large proportion of the sugar, the prevention of which destruction is the principal function of the vacuum pan. There has thus been instituted the custom of boiling down the sugar solution at a much lower temperature than that most favourable to its perfect crystallisation, and only imparting this temperature for a short time at the end of the process by means of the heater.

A diagrammatic representation of the several stages of sugar refining until it reaches the reheater is shown in Figs. 80 and 81.

THE FILL-HOUSE

The moulds for forming loaf sugar are arranged in a capacious building. They are placed on wooden supports, which keep them perfectly in line in the piece.

The moulds (Fig. 82), the profile of which represents a sugar loaf, such as it is known to every one, are made of iron, painted on the

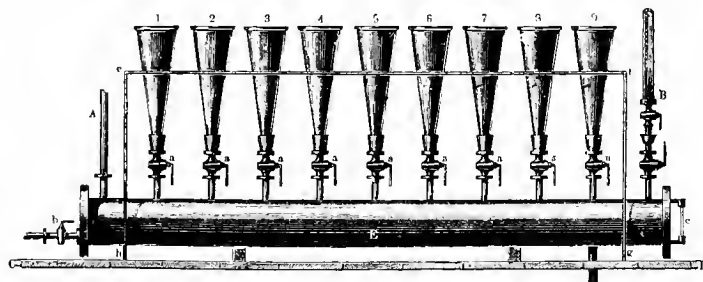


FIG. 82.—Sugar-loaf moulds for draining sugar loafs by suction.

one side with a sort of enamel; at their apex is a hole which is closed by a wooden peg. All these moulds are placed empty in their supports, with the apex pointing downwards.

When the boiled mass is run into the reheaters the workmen bring their large beaked basins, shaped something like coal-scuttles, which they carry by straps, and fill them by opening the valve, and then discharge them by pouring them into the moulds, which are thus filled one after the other with the hot boiled mass. The work of these men is often done by machinery. A trough running on wheels above the moulds fills a whole row at a time, and so on from row to row in succession.

The moulds thus filled are soon covered with a solid layer of crystallised cooled sugar. A workman provided with a large wooden knife or spatula, technically known as a hauling knife, breaks the crust and energetically stirs the mass so as to distribute the crystals until the loaf has gained a certain consistency. Sugar cones over

14 lbs. in weight are not considered loaves in commerce, but are known as lumps, titlers, etc. These are not generally subjected to the process of hauling. It is then left to itself. Care is taken to keep the room at a constant temperature of 30° to 35° C. (86° to 95° F.), and especially to avoid air currents or draughts.

The moulds and their contents are left to stand at rest for a period of at least twelve hours, except as far as having been stirred in the face during the early portion of this period, so as to make sure that the crystals form and settle in a regular and uniform manner. During this period of repose the crystals as they form settle, and moreover, as the superincumbent syrup cools, the sugar which it deposits acts as a binding agent to cement the mass into one homogeneous whole.

But as the solution of sugar which on evaporation and boiling down yielded the boiled mass was far from being pure, and was moreover more or less coloured, it results, as a matter of course, that all the impurities have been concentrated to a small bulk in the boiled mass, or rather, and in less bulk still, in that portion which still remains in the liquid form. This impure liquid is known as the "green syrup," and must be eliminated from the crystals forming the loaf before the latter can be dried and put on the market for sale. With this end in view, the loaves are transferred by lifts to an upper storey, and, first of all, the wooden plugs which have been used to stop up the apex of the moulds are removed and a pricker or brad-awl is forced up a few inches into the crystalline mass, so as to make a channel through which the impure syrup may drain itself away. The moulds, unstoppered and pierced or primed in the manner indicated, are now placed apex downwards on racks or tables, troughs pierced with holes of the same shape as the moulds underneath, which are inclined troughs or gutters almost on a level with the floor, which collect the whole of the syrup from many hundreds or thousands of these cones and conduct it into one general reservoir. Here it may once more be usefully observed that, no matter how white or pale the original sugar liquor was before boiling, that operation, even when conducted with the utmost care by means of the vacuum pan at the lowest possible temperature under the most favourable circumstances, brings in its train a certain amount of destructive action and consequent darkening in colour. It will not therefore be a matter for surprise that not only is the syrup which flows from the pierced cone not perfectly colourless, but the sugar itself from which it drains is not completely white, even though the coloured syrup which occupied the interstices between the crystals has been drained away. Enough syrup, indeed, remains adhering to the crystals to colour the whole mass a dark yellow or even brown. Each crystal is, in fact, surrounded by a coating of coloured substances, so obstinately persistent that nothing less

than a washing of the crystals will suffice to remove it. This washing process is performed after the loaves have drained for about a week.

Many modifications of the washing process have been tried from time to time. The claying process is the oldest and perhaps not the least effective method. This consisted in the application of *clay magma* to the face of the cones, just as is done in the manufacture of clayed sugar from the original cane juice. This method of washing is known by the name of *claying*, and may well be designated *claying properly so called*, to differentiate the process now universally adopted in sugar refineries, which has also been designated as claying, although no clay whatever is used in the process. The "*claying*" process consists in breaking the upper crust of the face or base of the sugar cone, which is very hard, and in applying to the fresh exposed surface a thick magma or paste of sugar and water. The action of this superimposed layer is similar to that of wet clay. A certain proportion of the concentrated saccharine fluid gradually percolates through the interstices of the loaf, driving the coloured syrup before it. It will be at once seen that, by repeating this process sufficiently often, the sugar cone would eventually become perfectly white.

In actual practice, however, that is not the course pursued: the so-called *claying* process is merely a preliminary to the subsequent process of *liquoring*.

The rationale of the liquoring process depends upon the sufficiently apparent principle that sugar at any given temperature is insoluble in water which has already been saturated with sugar at that temperature. A saturated solution of pure sugar is prepared of a density of 1.091 (33° Baumé; 18°.2 Twaddell), and stored on the top floor of the refinery under the name of "*fine liquor*" or "*magma liquor*." This liquor, not being subject to fermentation, may be stored for an indefinite period. The liquoring process follows the operation of claying. The "clay," or paste of sugar and water, was superimposed on the face or base of the sugar cone. By this time it will have driven a certain amount of the coloured syrup before it, and the sugar loaf will gradually have become dry. The workman now smooths and evens the base of the sugar loaf with his bottoming trowel. He then runs on magma liquor to the depth of some inches, and allows it to percolate through the whole cone. The process of liquoring is an interesting one. The expulsion of the dark-coloured syrup is not the only result attained: the sugar left behind increases in weight by the segregation of fresh crystals coming from the *fine liquor*. Consequently the loaf actually increases in weight instead of decreasing. It is stated that for some reason or other the combined processes of *claying and liquoring*, as now practised in sugar refineries, does not succeed except on sugars which have been prepared by the vacuum pan. Hence in those countries where open-pan boiling is still in vogue the process of *claying* with *clay* is still practised.

A single liquoring operation seldom suffices to purify a sugar cone. Several successive liquorings are given, almost always two, occasionally three, but seldom more than three. The progress of the process is judged from time to time by knocking a loaf out of its mould from a lot under treatment, and inspecting and observing how the percolation is proceeding. Should the crystalline mass present a satisfactory appearance, the cleansing process is delineated by a sharp distinct line. On the other hand, if the boiling process has been unsatisfactorily conducted, or if the sugar was of bad quality to start with, then the cleansing goes on in a very irregular manner.

In any case, this washing of the crystals is continued until the loaf is uniformly white throughout its whole mass. A sugar cone may be ascertained to be free from coloured syrup that is arrived at the stage of the process technically known as neat by the appearance of the liquor which percolates from the perforated apex of the mould. A couple of days is now allowed for complete draining. As percolation begins to cease, the sugar cones are loosened in their moulds by knocking the moulds against a solid wooden block. When the cones have completely ceased dripping and percolation is finished, the faces of the cones are trimmed or dressed—that is, the loose sugar on the face is cut off, either by means of a rotating blade called the facing machine, or by the triangular instrument already described, so that all the loose, irregularly aggregated, and imperfectly crystallised sugar on the face of the loaf is removed.

The cones are now ready to be knocked out of their moulds and subjected to further treatment, such as having their wet apices or noses cut off. But the treatment varies with the dimensions and other characters of the sugar loaves themselves. Those cones which are over 16 lbs. in weight, and known in commerce as lumps or titlers, have, as just stated, simply their apices truncated by a knife struck by a wooden mallet. But the apices of real sugar loaves, in the proper acceptation of the term, are retained, or, rather, new apices are affixed to them by means of a conical cutter attached to a kind of turning-lathe. This tool is known in the trade as the “nosing machine.”

Nothing further requires to be done to the loaves except to dry them. The next step, therefore, whether in the case of loaves, lumps, or titlers, consists in wrapping them in paper. The sugar cones in their paper envelopes, so as to prevent them from getting dirty, are then deposited on trellised shelves in a room capable of holding about 4000 loaves, called the stove, and heated by steam to about 54° C. (130° F.). The loaves are kept in the stove for a period of four to six days, after the expiry of which time they are equalised at face and apex, wrapped in paper, tied, and are then ready for sale.

All these operations take a long time and require an enormous space, as all the draining syrups are reboiled and are used in the manufacture of inferior quality sugars of the same form as the "firsts" sugars.

Facilitating percolation and drainage of sugar loaves by suction.—When the drainage syrup issues colourless, the moulds are subjected to suction. The operation is conducted as follows. Connected with the air-pump are horizontal pipes furnished with conical caps with indiarubber fittings, on which the moulds are fixed air-tight with the apex downwards. A vacuum made in the pipe by a pump, sucks the syrup, and removes the last dregs of the now useless cleansing liquor.

Sugar tablets.—There is another method of refining sugar, consisting in the manufacture of sugar tablets.

In the case of a round sugar loaf it must be cut into round tablets, perpendicular to the axis, and then broken into regular lumps for sale. All the lumps formed by the circumference are irregularly shaped and of less value. The question therefore was: why not make quadrangular tablets which would only produce regular cubes without waste? That is the reason why attempts have been made to change the classical form of the sugar loaf and to replace it by regular tablets, which only require to be broken parallel with the sides to produce uniform cubes.

The manufacture of sugar in tablet form has assumed enormous developments. In certain refineries attached to the French beet-sugar factories they only make this kind of sugar. The first stage of the refining process is the same. The change does not take place till after the boiling, when, instead of pouring the hot mass into moulds, it is poured into the ordinary rectangular moulds placed on a truck which is brought directly underneath the reheater to be filled. These moulds are then placed in turbines. They must therefore be of such a shape as will allow this to be done easily. They consist of rectangular wrought-iron frames, 8 inches across, and of the same height as the drum of the turbine. These frames are divided into compartments of about an inch thick by sheet-iron partitions which move on slides. To fill these frames, several are piled together, one on the top of another, keeping them firmly together, and the boiled mass is then run in. They are then left to cool, after which the frames are separated and firmly fixed in a turbine specially designed for the purpose (Fig. 83), which is turned for twenty minutes. The first molasses, or green syrup, flows away by itself under the action of the centrifugal force. Then by means of a measuring vessel the necessary quantity of cleansing liquor is introduced to the centre of the turbine, to purify it. After another twenty minutes the liquor has penetrated the blocks of sugar. The revolutions are kept up for another twenty minutes, and the operation is finished. The frames are then dismantled, the tablets extracted, and placed in the stove

for about eight hours; finally, the tablets are broken by special machines and packed in boxes for sale.

This process, more or less modified, improved, and adapted by the different inventors * who have contributed to render it practicable, is a very rapid one, and yields excellent results. Nevertheless, the sugar loaf yields more brilliant and more beautiful sugars, and will always preserve its value and prestige.

Raw syrup or molasses.—The residual green syrup or molasses remains to be treated or sold separately. In France, in the present state of the fiscal legislation of that country, molasses are no longer

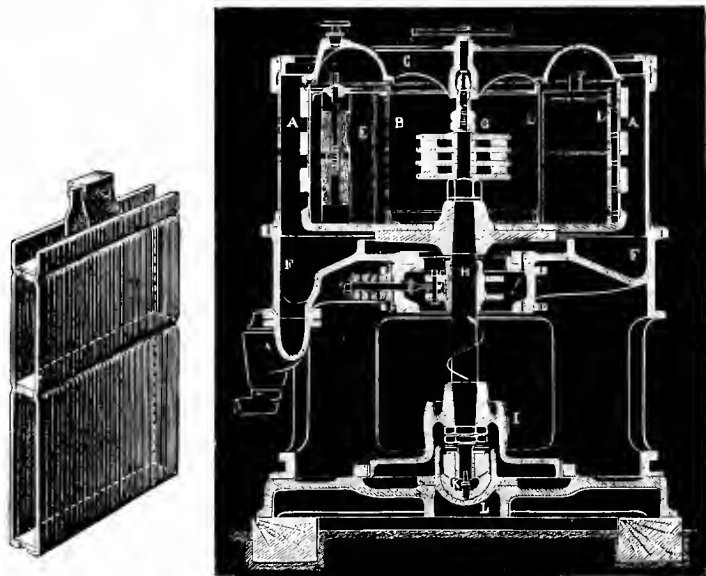


FIG. 83.—Sugar-tablet turbine, for use in manufacture of cube sugar.

sold. They are again wrought up for sugar. A long time ago they were osmosed. Then they were treated with baryta, a process which was abandoned on account of the difficulties connected with the regeneration of the carbonate. They then fell back on strontia, using the same processes as in beet-sugar works. At the present time Steffen's process is again about to be used in French refineries, so as to abolish molasses as far as practicable. At the balancing of accounts or stocktaking the waste in manufacture is very small, much less than that allowed by the Legislature; that is what constitutes the bounty of the French sugar refiner.

* See Abridgment of Specifications for Sugar Patents, for further details.

CHAPTER XIII

THE CHEMISTRY OF MOLLASSES, INVERT SUGAR, AND RAFFINOSE — THE ANALYSES OF MOLLASSES AND TABLE SYRUPS — CLERGET'S TABLE

THE CHEMISTRY OF MOLLASSES

MOLLASSES or treacle is the liquid, uncrystallisable, viscous mother-liquid from which sugar has been crystallised. Molasses from cane sugar contains about 35 per cent. of saccharose (crystallisable sugar), whilst that from beetroot contains 50 per cent. These relatively large amounts of real sugar cannot be crystallised, on account of the great concentration of impurities by the elimination of several previous crops of sugar crystals from the liquid in question. Moreover, it requires the presence of a large quantity of water to keep these impurities in solution; so much so that any attempts to further concentrate the mass cause it to become so thick and consistent, that, even if it were possible for sugar to crystallise in such thick sticky media, it would be impossible to separate the crystals from such a sticky and viscous mother-liquor.

Gill gives the following analyses of molasses from both sources :—

LXXIII.—CHEMICAL COMPOSITION OF MOLLASSES.*

	Molasses from Colonial Sugar.	Molasses from Beetroot Sugar.
	Per Cent.	Per Cent.
Cane sugar	35·0	49·0
Glucose	32·0	3·0
Ash	5·5	12·5
Organic matter other than sugar (gum, etc.)	9·5	15·5
Water	18·0	20·0
	100·0	100·0

* See also Tables LXXIV. and LXXV. p. 388.

The molasses from cane sugar is much superior to that from beet-root sugar. From about 20 to 35 per cent. of the impurities present in cane sugar consist of sugar which has changed during the process. The sweetening capacity of this sugar is almost equal to that of crystallised cane sugar. It consequently commands a certain value on the market. Molasses from beetroot sugar, however, has a nauseous bitter taste, owing to its abounding in saline impurities.

A glance at the above analyses will suffice to show that beet molasses is highly charged with impurities. It contains, in fact, all the concentrated impurities of the beet, and the impurities present in all the ingredients used in its treatment, such as lime and water, both of which deposit their soluble salts in it. Beet-sugar molasses is, in fact, a highly complex organic and inorganic product of a gluey nature, a fusty smell, and very bad taste.

Determining the density of molasses by the hydrometer.—Owing to its great viscosity, it is not easy to determine the density of molasses. Baumé's or any other hydrometer only gives approximate results, and can only be used for getting some rough idea thereanent.

By a 50 c.c. flask.—A more exact method is to fill the whole of the bulb portion of a previously tared 50 c.c. flask with syrup, by running it in through a funnel with a long stem; it is then weighed to ascertain the weight of the molasses. The flask is then placed under a Mohr's burette containing water, and the flask is filled to the mark with water from the burette. The volume of water added is deducted from 50 c.c., and the difference represents the volume occupied by the molasses, the weight of which is known. The two elements for calculating the density are thus ascertained: $D = \frac{W}{V}$.

ANALYSIS OF MOLASSES

The analysis of molasses is troublesome. If dissolved in water it yields a black liquid difficult to decolorise, which entails many precautions. Yet its analysis is necessary, because it contains on an average 50 per cent. of sugar, and it is sold by its percentage of the latter ingredient to the distiller; and it is by its analysis that the distiller is guided in his treatment of it, so as to convert it into alcohol.

Five times the weight of the quantity of molasses to be used in the saccharimeter employed—say 130·24 grammes for the German instrument and 81 grammes for the French instrument—are weighed into a capacious nickel capsule with a wide spout. Weighing is facilitated by the use of a glass rod, so as to add or remove a small drop of molasses as may be required. A small quantity of hot water is run into the capsule, stirring the mass with a glass rod. The molasses partially dissolves. The liquid is decanted into a graduated flask of 500 c.c. capacity, supporting the beak of the capsule against

the mouth of the flask so as to avoid using a funnel. Hot water is again added to the remainder of the molasses to dissolve a fresh portion, which is likewise poured into the flask, and so on until the whole of the molasses weighed out is dissolved.

The solution will then amount to about 300 c.c. Towards the end the capsule is rinsed out with a jet of hot water, and the wash-water is added to the principal solution. The whole is well agitated by rotating the flask, and brought to the surrounding temperature; 25 c.c. of basic acetate of lead are then added—that is to say, the volume strictly necessary to produce the precipitate. The volume is made up to 500 c.c., agitated and filtered, and 100 c.c. observed in the saccharimeter.

Should the filtered liquid be too dark to be exactly observed in the saccharimeter, it is decolorised with a little fine, dry, specially prepared animal charcoal, previously exhausted with hydrochloric acid, exactly neutralised with alkali, washed, and dried. The black is introduced into the filtered liquid along with a little zinc dust, stirred with a rod, and filtered after fifteen or twenty minutes. The filtered liquid is then so far decolorised as to be examined in the saccharimeter. A confirmatory volumetric test is made by means of Fehling's solution. As it may happen that the indications of the saccharimeter are not always exact, from the fact that the uncrystallisable sugar in the molasses may be optically active, and thus of themselves so act on polarised light as to vitiate the ordinary results, in that case direct polarisation cannot show the percentage of crystallisable sugar in a positive manner. The latter must therefore be estimated by optical inversion. This method depends on the fact discovered by Clerget, that when crystallisable sugar is inverted by hydrochloric acid the solution of invert sugar which results deviates the plane of polarised light to the left, and that of a normal solution showed 100° to the right, the same solution inverted would indicate 44° to the left at the temperature of $0^\circ \text{ C.} = 44 - \frac{T}{22}$ at the temperature T, so that the deviation would be nil at 88° C.

Generally, on account of other sugars, therefore, it is necessary to make an inversion in the following way. The direct observation is made in the usual way, the remainder of the liquid is filtered, and a flask graduated at two points, 50 and 55 c.c., is filled to the 50 c.c. mark, and 5 c.c. concentrated gumming hydrochloric acid, sp. gr. 1.188 = 38 per cent. HCl, are added (that is, up to the 55 c.c. mark). The flask is shaken to make the liquid homogeneous, and inserted in a water bath kept at the temperature of 68° C. A thermometer is inserted in the flask, which is shaken so as to bring its contents uniformly to 68° C. , which takes about five minutes, after which the flask is kept at the same temperature of 68° to 70° C. for ten minutes longer, when the flask is withdrawn and cooled by immersion in

water ; when it has cooled down to 20° C. the thermometer is withdrawn, and 0·5 to 1 gramme of animal charcoal added, stirred, and filtered rapidly. By the addition of the 5 c.c. of hydrochloric acid the volume of the sugar solution is increased by one-tenth ; the optical properties of the latter are consequently weakened to the extent of one-tenth. In order to compensate for this weakness, a tube of 22 centimetres in length is taken instead of a 20-centimetre tube.

Moreover, the temperature, which is almost without influence on the rotatory power of cane sugar, very perceptibly modifies that of invert sugar. That is why the 22-centimetre tube carries a thermometer which indicates the temperature of the sugar solution at the moment of the experiment.

The liquid is therefore transferred into a tube 22 centimetres in length, bearing a thermometer ; the tube is then placed in the apparatus, and the operation conducted as before. The index of the compensator is no longer moved towards the left, but rather to the right ; the number on the graduated scale is then read. This number is added to that obtained before inversion, and the richness in crystallisable sugar, which corresponds to this total at the temperature of the observation T, is then found by consulting *Clerget's* tables. If the index, after the action of the acid on the sugar solution, was still to the left of zero, it will be necessary to subtract these two numbers instead of adding them.

In default of *Clerget's* tables, very approximate results may be obtained by means of the following formula :

$$x = 1.635 \times \frac{200 S}{288 - T}$$

in which S represents the sum or difference of the numbers given by the instrument before and after inversion ; x , the number of grammes of sugar contained in 1 litre of the solution ; and T, the temperature of the sugar solution.

Landolt modified *Clerget's* formula thus :

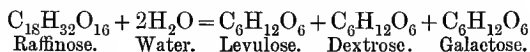
$$C = \frac{200 S}{284.8 - T}$$

in which C = crystallisable sugar ; S, the sum of the two saccharimetric readings before (P) and after (P_1) the inversion, without taking the negative sign of the latter into account ; and T, the temperature of the last reading in ° C.

The value of C calculated in this way is compared with the result of the direct observation P. If C is greater than the direct observation, the sugar tested contains inverted sugar. On the contrary case, there are dextro-rotary substances present.

RAFFINOSE OR MELITRIOSE

The molasses of the sugar refinery contains a *triose* sugar—*raffinose* identical with melitose ($C_{18}H_{32}O_{16}$), the sugar extracted from Australian manna. It may be separated from molasses by means of alcohol, in which it is less soluble than cane sugar. Raffinose is inverted into three glucoses, when its solution is heated with diluted sulphuric acid—



ESTIMATION OF RAFFINOSE

A solution of raffinose is dextro-rotary; it does not reduce *Fehling's* solution. Certain kinds of sugar, especially those extracted from molasses by the saccharate process, frequently contain raffinose, which, in consequence of its high dextro-rotary power (at 20° C. $p = 0 - 10 + 104 \cdot 5$ (constant) being greater than that of crystallisable sugar, imparts an abnormal polarisation thereto. The total of the usual estimations therefore occasionally exceed 100 per cent., without taking into account the undetermined substances. Moreover, even in cases where the total sum of the percentages of the different estimation is less than 100, the excessive amount of the crystalline sugar is proved by the results of the optical inversion.

However, the formula given above does not suffice to rectify the percentage of crystallisable, and still less does it admit of the amount of raffinose being deduced from it.

But both the crystallisable sugar and the raffinose may be easily ascertained by the use of *Creydt's* formula, deduced from direct experiments on mixtures of chemically pure samples of the two substances in known proportion.

Creydt found that after a solution of cane sugar indicating +100° of the saccharimeter had been inverted, as previously described, the inverted solution at a temperature of 20° C. showed a deviation of -32°. A solution of raffinose, on the other hand, showing +100° by the saccharimeter, will yield after inversion a deviation to the right of 50°·7 at 20° C. A mixture of the two substances, in definite proportions, similarly treated should yield an intermediate rotation.

Let—A = Polarisation before inversion,
 B = ,, after inversion at 20° C.,
 C = Difference between A and B,

the amount the polarisation has been reduced: the percentage of *crystallisable sugar* S, and of *raffinose* R, may then be calculated from the following formulæ:

$$(a) S = \frac{C - 0.493A}{0.827} = 0.613A - 1209B$$

$$R = 1.017A - \frac{C}{21.98}$$

Of course this calculation is only applicable to those cases where only these two optically active substances are present. In the case of more complicated mixtures, the result is less exact the larger the quantity of other substances present.

A German authority gives the following formula :

$$R = \frac{I + 51.24 \times P}{0.8390}$$

in which P = polarisation before inversion, and I = polarisation after inversion.

Estimation of raffinose in sugar products—(a) First products.—100 grammes are shaken in a stoppered, narrow-necked 250 c.c. flask with 150 c.c. of commercial wood spirit, previously mixed with a few drops of potash alum solution to neutralise the alkalinity of the sugar. The precipitated sulphates also carry down colouring matter. A 100 c.c. flask is filled to the mark with the solution, 40 c.c. removed by distillation, 20 c.c. of water added, and lead acetate as long as a precipitate forms. A little moist aluminium hydrate is then added, the flask filled to the mark with water, 5 c.c. of hydrochloric acid (36 per cent. HCl) added, and heated for ten minutes at 68° C., cooled rapidly to 20° C., and polarised in the 200-millimetre tube. The value of raffinose calculated from the equation, and multiplied by 1.5 = the percentage of raffinose within 0.05 per cent. of raffinose, may be estimated in this way, but to extract such small amounts some potassium acetate must be added to the solvent.

(b) For solid after-products.—The raffinose in these is only dissolved very slowly, incompletely by methyl alcohol, and a modification of the method is therefore necessary. 30 grains of raw sugar are treated in a graduated flask with 6 to 9 c.c. of water and enough potash alum solution to neutralise the alkalinity of the sugar. The sugar is then dissolved by heat, and about 120 c.c. of wood spirit added, with continual shaking. After cooling, it is made up to the mark with wood spirit, a little powdered sugar added, and the flask shaken frequently during one hour. After from 10 to 11 c.c. of wood spirit have been added to correct for the volume of sugar separated, the liquid is filtered and the estimation continued as directed under (a). The value for raffinose found, multiplied by 5, gives the percentage of raffinose. These estimations are accurate within 0.1 per cent. of raffinose.

(c) Molasses.—On account of the solubility of molasses in methyl alcohol or wood spirit no concentration of the raffinose can take place, and the method is therefore very similar to the German inversion

method. 12 grammes molasses are mixed in a 100 c.c. flask with 12 grammes water, and the amount of potash alum solution necessary for neutralisation dissolved, and wood spirit added to the mark. It is then shaken, filtered, and the filtrate analysed as under (a). The value found, multiplied by 12.5, gives the percentage of raffinose present. In America the estimation of raffinose in raw sugars is made by a method only slightly differing from the German one.

The Austro-Hungarian Centralverein obtained several imported and a high-grade home-produced table syrup, and had them examined by the methods of Herzfeldt and his followers. The following results were obtained :—

LXXIV.—TABLE SYRUPS—ANALYTICAL DATA.

	Imported Table Syrups.				Domestic Syrup.
	I.	II.	III.	IV.	V.
Specific gravity at 17°·5 C.	1·4233	1·4173	1·4344	1·4309	1·4181
Degrees Balling	81·10	80·22	82·74	82·23	80·33
Direct polarisation	38·50	37·80	36·05	32·90	52·50
Polarisation at 20° C. after inversion	16·1	17·0	16·50	16·0	17·0
Reducing sugar, determined as invert sugar according to Preuss	25·50	21·56	28·34	21·67	6·47
Total solids	74·13	71·04	74·03	74·28	77·55
Ash	3·75	5·07	4·68	6·94	9·74

The following composition is calculated for the syrups from these analyses :—

LXXV.—TABLE SYRUPS—COMPOSITION.*

	I.	II.	III.	IV.	V.
Water	23·87	28·96	25·97	25·72	22·45
Cane sugar	41·91	41·31	39·60	37·30	52·45
Invert sugar	7·09	10·32	10·44	12·94	6·47
Optically inactive sugar	18·41	11·24	17·90	8·73	0·00
Ash	3·75	5·07	4·68	6·94	9·74
Organic matter not sugar, by difference	2·97	3·10	1·41	8·37	14·89
	100·00	100·00	100·00	100·00	100·00

* See Table LXXIII. p. 382.

Optically inactive sugar is here a mixture of glucose and invert sugar, in which the right-handed rotation of one is exactly equal to the right-handed rotation of the other. These analyses show that the domestic syrup is nothing more than a beet-sugar molasses, whilst the imported syrups are a better grade of colonial cane-sugar molasses. The taste and general appearance of the imported syrups showed them to be colonial molasses, whilst the home-produced table syrup did not taste much better than common molasses.

CHAPTER XIV

THE ANALYSES OF RAW SUGARS, COMMERCIAL GLUCOSE, AND GLUCOSE SYRUPS

ANALYSES OF RAW SUGAR

THE commercial analyses of raw sugar comprise the following determinations :—

1. Moisture.
2. Crystalline or cane sugar.
3. Uncrystallisable sugar, or fruit-sugar glucose.
4. Ash.
5. Organic matter.
6. Yield of refined sugar.

1. *Estimation of moisture.*—Moisture is determined on 5 grammes of substance, which is dried at 105° C. until of constant weight—about two hours for normal sugar. The ash determination is often made on that used for the moisture, so as to avoid a fresh weighing of 5 grammes.

2. *Cane sugar.*—The amount of sugar corresponding to the saccharimeter to be used—namely, 16·19 grammes for the French instrument and 26·048 for the German instrument—is dissolved in 50 to 70 c.c. of water. The sugar may be dissolved in different ways, namely, by weighing it in a platinum basin and dissolving it in the same dish ; or it may be transferred to a test-glass on foot, the basin being rinsed with a little water, which is afterwards used to dissolve the sugar in the glass. In any case, the lumps of sugar difficult to dissolve are crushed with a glass rod (dubbed out into a knob at the end), which is also used to stir the liquid, and thus facilitate solution. Solution accomplished, the liquid is cautiously run into a 100 c.c. flask, the glass rod being placed against the lip of the platinum dish, or test-glass on foot, during the operation, which with the rod are washed with water to remove all trace of sugar. The wash-waters are collected in the 100 c.c. flask, which in this way is filled to about three-quarters of its weight. Some chemists prefer to operate in a different way. The sugar is weighed on a small piece of sheet-aluminium, in the form of a gutter, then by means of a nickel silver funnel the sugar is run directly into the 100 c.c. flask by the

aid of a jet of water, which at the same time rinses the funnel. When the sugar is greasy it often adheres to the aluminium foil used in weighing. It is, however, very easy to remove the adherent crystals by means of a flat brush made by trimming a goose quill, and the aluminium foil remains dry and is ready for use immediately afterwards for the next experiment. The flask is then three-quarters filled by the addition of water, after which a rotary movement is imparted to it to dissolve the sugar. Transferring and washing are thus avoided, which is an advantage when a series of similar analyses are to be made. But solution is effected more slowly. The sugar being completely dissolved, a little basic acetate of lead is added, 0.1 to 1 c.c., according to the depth of colour of the solution, so as to precipitate colouring matters, and water is then added to fill the flask almost to the mark, air-bells dispersed with a drop of ether, and the solution then made up to the mark with water. The turbid liquid is shaken to render it homogeneous, filtered, and examined directly in the polarimeter for the percentage of saccharose in the raw sugar.

In France polarimeters of the Laurent type are used, the normal official weight of which is 16.19 for 100 c.c. of the metric system, whereas in Belgium the German instruments of the Venzke type are used, the normal weight of which is 26.048 grammes for 100 c.c. of Mohr's system (say 26 grammes for 100 c.c. metric system, see p. 15).

3. *Uncrystallisable sugar*.—The presence of this sugar is confirmed by a qualitative test with Fehling's solution; in which case the liquid remaining from the polarimetric examination is freed from its excess of lead by mixing it with 5 c.c. of a saturated solution of carbonate or sulphate of soda, and filtering. The dilution of the liquid may be avoided by adding finely powdered dry sulphate of soda. The filtered liquid is run into a burette and $\frac{1}{2}$ to 1 c.c. of Fehling's solution titrated therewith until the liquid floating on the precipitate of cuprous oxide becomes colourless. By titrating 1 c.c. of Fehling's solution, corresponding to 0.005 gramme of glucose, the quantity of glucose in 100 c.c. of the sugar solution (x) is first found, then that in 100 grammes of the sugar (y) by the following proportions:—

$$0.005 : n = x : 100 ; x = \frac{0.5}{n} ;$$

$$\frac{0.5}{n} : \text{normal weight} = y : 100 ;$$

$$y = \frac{50}{n \times \text{normal weight}}.$$

In Germany the gravimetric method is used, weighing the metallic copper obtained by the reduction of the protoxide. In the case of sugars containing more than $1\frac{1}{2}$ per cent. of glucose, Meissl's table (XVIA. p. 47) is used, whilst Herzfeldt's table (XV. p. 46) is used for those which contain less, always working on 10 grammes of sugar

dissolved in 50 c.c. of liquid. So as to utilise the solution remaining from the polarisation (26·048 grammes in 100 c.c.), 76·8 c.c. of the liquid is drawn by means of a special pipette and a saturated solution of carbonate of soda added, and the bulk brought to 100 c.c. It is filtered and 50 c.c. of liquid drawn off, containing exactly 10 grammes of sugar.

4. *Estimation of ash.*—5 grammes of the sugar are weighed into a tared platinum capsule and moistened with sulphuric acid, and incinerated in a muffle furnace at a dull red heat; the increase in weight gives the sulphated ash in 5 grammes of the sugar, which, multiplied by 20, brings it to per cent. As the weight of the ash rarely exceeds 0·2 gramme, Sidersky uses a small invariable weight of 4·8 grammes, which he uses in every weighing of ash, only the weights varying between 0·1 and 0·2 gramme, according to the richness of the sugar in ash, have then to be added, and the operator need only trouble himself with these weights, as the 4·8 grammes remain constant.

Determination of the true ash in products of cane-sugar manufactories, incinerated with sulphuric acid.—The determination of the ash in products of sugar manufactories is easily made according to the method of Scheibler, known as sulphuric incineration. It was proposed in 1865, in order to circumvent the inconveniences in the direct incineration proposed by Pelegot in 1851, and carbonisation with titration of the aqueous extract of the ash proposed by Dubrunfaut in 1832. Scheibler, recognising that sulphuric incineration gave a greater weight of ash than direct incineration, and that this difference might be calculated, proposed to apply to the sulphated ash a coefficient for correction which he fixed at 0·90, which to-day is generally adopted. However, in 1870 Dubrunfaut drew attention to the fact that this coefficient of 0·90 could only be considered as a mean value, which might vary sensibly from the truth in many cases. He then proposed to add to the sugars finely powdered baker's charcoal, or, better, platinum black. Dubrunfaut gives the following experiment. A sugar incinerated with spongy platinum gave 1·724 per cent. of ash; by direct incineration, 1·730; by sulphuric incineration, corrected by the coefficient 0·9, it gave 1·791. The first two results agree, giving a mean of 1·727, while in the third result the quantity obtained from a weight of sulphated ashes of $\frac{1\cdot791}{0\cdot9} = 1\cdot99$. A true coefficient to convert the sulphated into true ash would thus be $\frac{1\cdot727}{1\cdot99} = 0\cdot868$, and the coefficient 0·90 would be too high. This was Violette's conclusion, who suggested 0·8 instead of 0·9 as being more exact for raw sugars excepting "firsts," for molasses, diffusion liquors, and the beet itself. For "firsts" sugars the coefficient should not be more than 0·7.

Estimation of true ash in sugars and syrups without sulphuric acid.

—Oxide of zinc is used, which has the following qualities to recommend it: It is white, infusible, forms only a single oxide, is easily obtained in impalpable powder, is not hygroscopic, and it yields its oxygen as easily as the oxide of copper in the presence of organic matters, and in a measure indefinitely, because the zinc, reduced to a metallic state, is reoxidised immediately, and is susceptible, therefore, of furnishing a new quantity of oxygen to the substance until its complete combustion. The operation is conducted as follows:—

In the case of sugar, 5 grammes are weighed in a platinum dish, to which are added 0.05 gramme oxide of zinc and a few drops of distilled water. The carbonising is then carried on with due precaution over a Bunsen burner. The mass, on melting, absorbs evenly the oxide of zinc. After the carbonising is completed the capsule is placed in a muffle, heated to low redness, and the incineration is completed after three-quarters of an hour, or an hour at most. The ash is weighed and the 50 milligrammes of oxide of zinc subtracted. The capsule should be dried in a dessicator and weighed as rapidly as possible, as the carbonate of potash present is highly hygroscopic.

For syrups the operation is carried on in the same manner, except sometimes it is preferable to weigh only 3 grammes instead of 5. The capsules of platinum do not seem to suffer anything from the presence of the oxide of zinc, but porcelain dishes can be used instead.

Estimation of ash in sugar by incineration with benzoic acid.—To avoid the correction of $\frac{1}{10}$ as proposed by Scheibler, and $\frac{1}{5}$ as proposed by Girard and Violette, when sugars are burnt with H_2SO_4 , Boyer suggests incineration with benzoic acid as giving the real quantity of mineral matter without correction. The benzoic acid is dissolved in alcohol of about 90 per cent., 25 grammes of the acid to 100 c.c. of alcohol. 5 grammes of the sugar are weighed in a capsule and moistened with 1 c.c. water. The capsule is heated slowly in order to caramelize the sugar without carbonising it; 2 c.c. of the benzoic solution are next added, and the capsule warmed until all the alcohol is evaporated; the temperature is then increased until the sugar is converted into carbon. The decomposing benzoic acid produces abundant vapours, which render the mass extremely porous, especially if a circular motion be applied to the capsule. The slow heating is continued until all the benzoic acid is volatilised. The carbon obtained is voluminous and of a brilliant black colour. The incineration is finished in a muffle at a low red heat. The capsule should be weighed quickly when taken from the dessicator, to avoid the absorption of water by the alkaline carbonates. Benzoate of ammonia may be used instead of benzoic acid, and the freedom of both acid and ammonia salt from residue on ignition should be tested. Besides giving the mineral matters directly, by this method their composition may also be determined—a rather important item.

5. *Organic matter*.—The organic matter is estimated by difference.

6. *Yield of refined sugar*.—The yield of refined sugar is estimated by deducting from the polarimetric result twice the weight of the uncrystallisable and four times the weight of the ash. That is the French yield. In England the coefficients 3 and 5 are used, that is to say, the glucose is multiplied by 3 and the ash by 5. (See Table LXVII. p. 332.)

ANALYSIS OF COMMERCIAL GLUCOSE

The percentage of dextrose in crystalline glucose is determined by polarising 20·4 grammes of substance in 100 c.c. of water prepared in the same way as in the case of raw sugar. The degrees read on Laurent's saccharimeter give directly the percentage of glucose. With the German polarimeter 16·41 grammes of substance are dissolved in 100 c.c. of water, and the result got in a 200-millimetre tube multiplied by 2, or the experiment is done in a 400-millimetre tube so as to get directly the percentage of dextrose.

It would not be practicable to use 32·8 grammes for 100 c.c., because the solution would be too concentrated and the rotary power would no longer be constant.

As in the case of other reducing sugars, in order to prevent bi-rotation the solution must be boiled before being brought to the measured volume.

ANALYSIS OF GLUCOSE SYRUPS

Composition.—These syrups generally contain dextrose, maltose, and dextrine, which are estimated by Wiley's process.

Wiley's process.—*Principles on which it is based*.—1. The specific rotary power of these three bodies being, dextrose = +52·74, maltose = +138·3, and dextrine = +194·8, a solution containing these three bodies will have a polarisation $P = 52·74 D + 138·3 M + 194·8 d$,—*D*, *M*, and *d* being the respective quantities of dextrose, maltose, and dextrine contained in the solution.

2. If the solution be treated by mercuric cyanide in excess, the dextrose and maltose are entirely destroyed and the dextrine alone remains intact. If the solution be now polarised, the deviation observed will give the quantity of dextrine present, and we get—

$$P = 194·8 d;$$

a formula from which we can easily calculate *d*.

Each degree of the saccharimeter being = 0·055 gramme, or = 0·089 dextrine, according as the weight taken for the polarimetric examination of cane sugar is 16·20 grammes or 26·048 grammes.

3. The difference of the two observations is—

$$P - P^1 = 52·74 D + 138·3 M.$$

The solution is then treated with Fehling's solution, which is reduced by glucose and maltose. The operation may be conducted either volumetrically or gravimetrically, and the whole is calculated as dextrose. As the reducing power of maltose is 0.65, that of dextrose being taken as unity, we get the total reduction—

$$R = D + 0.65 M.$$

By multiplying this last equation by 52.74 it becomes—

$$52.74 R = 52.74 D + 34.28 M,$$

which we subtract from 3 and we get—

$$(P - P^1) - 52.74 R = 104.02 M;$$

$$\text{whence } M = \frac{(P - P^1) - 52.74 R}{104.02}, \text{ and } D = R - 0.65 M.$$

The solution of mercuric cyanide used by Wiley contains per litre—

120 grammes of mercuric cyanide.

25 grammes potassic hydrate.

Wiley, in testing several American glucose syrups by this method, found that the dextrose varied between 23 and 42 per cent., the dextrine between 29 and 45, and the proportion of maltose between 1 and 19.

So as to get exact results, it is advisable to prepare a solution of 25 grammes of syrup of glucose in distilled water, which after boiling is brought to 100 c.c. The polarimetric observation is performed as indicated above, whilst the copper reduction (p. 26 *et seq.*) is effected on a dilute solution containing 1 to 2 per cent. of the syrup to be tested.

CHAPTER XV

THE ESTIMATION OF SUGAR IN WINE, DIABETIC URINE, MILK, HONEY, AND JAM

OPTICAL DETERMINATION OF SUGAR IN WINE

THE polarimetrical examination of wine is intended to discover certain sophistications, such as the addition of cane sugar, commercial glucose, etc. The process is conducted as follows.

I. *White wines*.—If the wine to be tested be a white wine, 100 c.c. of the wine are measured, and 5 c.c. of basic acetate of lead added and filtered after stirring; $2\frac{1}{2}$ c.c. of a concentrated solution of carbonate of soda are added to $52\frac{1}{2}$ c.c. of the filtered liquid to precipitate the excess of lead. The filtrate is examined in a 200-millimetre tube, and the degrees read, multiplied by 1.1 to make allowance for the dilution of the wine tested.

II. *Red wines*.—If it be a red wine, 10 c.c. of basic acetate of lead are added to 100 c.c. of the wine and filtered, and 5 c.c. of carbonate of soda solution added to 55 c.c. of the filtrate, the mixture shaken and again filtered. The filtrate is examined in the saccharimeter, and the degrees read, multiplied by 1.2 to get the polarisation of the original wine.

The wine may vary in its action on polarised light in three different ways—

1. It may be dextro-rotary.
2. It may be levo-rotary.
3. It may be optically inactive.

1. *The wine to be tested is dextro-rotary*.—It may contain saccharose or the other dextro-rotary non-fermentescible matter contained in ordinary commercial glucose. 50 c.c. are inverted with 5 c.c. hydrochloric acid in the manner already described, and a fresh polarimetric observation taken: if the inverted wine be levo-rotary, cane sugar is present; if, on the other hand, the rotation is still to the right, and exceeds that of 0.70 per cent. of glucose, it may be inferred that dextro-rotary substances from commercial glucose are present.

2. *The wine to be tested is levo-rotary*.—The left-handed rotation is due solely to inverted sugar formed by inversion of the natural sugar of the must, or by that of cane sugar added intentionally. If

a previous test has been made with Fehling's solution, it is first ascertained if the polarisation be approximately equal to that which results from the presence of a corresponding quantity of invert sugar. If the left-handed rotation is appreciably inferior to the result of the Fehling's solution estimation, non-inverted cane sugar or the dextro-rotary substances of commercial glucose are present. To demonstrate the presence of the latter, the wine is fermented; if the fermented liquid be dextro-rotary, non-fermentescible dextro-rotary substances are present, originating in a fraudulent addition of commercial glucose.

The wine to be tested is optically inactive.—This is the case with most natural wines. As the optical inactivity may sometimes be due to the simultaneous presence of dextro-rotary substances and invert sugar, so as to balance their different rotations, an inversion test is first made: if the inverted wine be levo-rotary, it may be concluded that cane sugar is present along with invert sugar. A fresh sample of the wine is then fermented, and if the fermented liquid is dextro-rotary the presence of commercial glucose is absolutely demonstrated.

THE UNITED STATES OFFICIAL METHOD

This is to be determined by Soxhlet's method. The presence of unfermented cane sugar is to be shown by inversion.

The wine should not contain more than 1 per cent. of sugar.

Preliminary test.—100 c.c. of wine accurately neutralised with normal soda solution is dealcoholised by evaporation to two-thirds, and the original volume restored with water (red wines are decolorised with pure animal charcoal); 25 c.c. of copper solution and 25 c.c. of Rochelle salt solution are heated to boiling in a deep porcelain evaporating dish; wine is added from a burette till the fluid, after two minutes' boiling, no longer has a blue colour. This gives an approximate estimate of the sugar present, and from this we can dilute the wine to very nearly 1 per cent. of sugar.

Final determination.—25 c.c. of wine, as in preliminary test, 25 c.c. of Rochelle solution, and 23 to 24 c.c. of diluted wine (approximately 1 per cent.), are heated two minutes to boiling, the precipitated copper allowed to settle, and filtered through a double filter. If the filtrate is greenish, it contains copper; if the filtrate is yellow, it is acidulated, and, after cooling, a little acetic acid is added and two or three drops ferrocyanide of potassium. If a reddish colour is formed, copper is still present, and a sufficient amount of sugar has not been added to reduce the 50 c.c. of Fehling's solution. The experiment is repeated, adding 1 to 2 c.c. more wine if copper was present in the filtrate; if no copper was in the filtrate, 1 c.c. less of wine. The experiment is continued till filtrates are obtained containing a difference of 0.1 c.c. of wine, one of which contains copper solution, and one not. The mean of the two contains the sugar equivalent to 50

c.c. of Fehling's solution ; 50 c.c. of Fehling's solution correspond to 0.2375 gramme of grape sugar.

Polarisation.—(1) The wine is decolorised with plumbic subacetate.

(2) A slight excess of sodic carbonate is added to the filtrate from (1). 2 c.c. of a solution of plumbic subacetate are added to 40 c.c. of white wine, and 5 c.c. to 40 c.c. of red wine, the solution is filtered, and 1 c.c. of a saturated solution of sodic carbonate added to 21.0 or 22.5 c.c. of the filtrate.

(3) The kind of apparatus used and the length of the tube are to be given, and the results estimated in equivalents of Wild's polaristrobometer with 200-millimetre tubes.

(4) All samples rotating more than $0^{\circ}5$ to the right (in 200-millimetre tubes, after treating as above), and showing no change or but little change in their rotatory power after inversion, are to be considered as containing unfermented glucose (starch sugar) residue.

(5) Rotatory power of less than $0^{\circ}3$ to the right shows that impure glucose has not been added.

(6) Wines rotating between $0^{\circ}3$ and $0^{\circ}5$ to the right must be treated by the alcohol method.

(7) Wines rotating strongly to the left must be fermented, and their optical properties then examined.

OPTICAL DETERMINATION OF SUGAR IN DIABETIC URINE

The estimation of sugar in diabetic urine is easily accomplished by the optical method in the case of but slightly coloured urine free from albumen. A 200-millimetre tube is filled with the bright and clear urine previously filtered, if need be, and the deviation read on the polarimeter used. With the French instrument the result is multiplied by 2.049 ; with the German, by 3.282, to get the sugar per litre of urine.

If the urine in question contains but little sugar, the polarimetric observation is made in a 40- to 50-centimetre tube, and the result divided by 2 and $2\frac{1}{2}$ respectively.

When the urine being tested contains albumen, which often happens, the latter must be eliminated before polarisation, the albumen being levo-rotary. To effect this, 100 c.c. of the urine are run into a porcelain basin, dilute acetic acid added, and the mixture brought to the boil. The albumen is soon deposited in large flakes, which collect on the bottom of the basin. The basin and contents are cooled, and water added to make up the original bulk, the solution filtered, and the liquid transferred to the saccharimeter.

It sometimes happens that the urine to be tested is too deep-coloured to get an accurate determination by the polariscope. With this end in view, 100 c.c. of urine are vigorously and repeatedly

ESTIMATION OF THE MILK SUGAR IN MILK 399

stirred with 2 or 3 grammes of finely pulverised animal charcoal, allowed to stand for some time, again stirred, filtered, and the decolorised liquid transferred to the polarimeter.

More frequently, however, the urine is decolorised by basic acetate of lead, which at the same time precipitates any small quantity of albumen which may be present. A flask graduated in two places, namely 100° and 110° c.c., is filled with urine to the 100 c.c. mark, and about 5 c.c. of basic acetate of lead added and the bulk made up to the 110° c.c. mark with water, the flask turned upside down and well shaken, the contents filtered and polarised. To make allowance for the dilution, the polarimetric reading must be increased 10 per cent.

The accompanying table shows for each degree of the saccharimeter the corresponding amount of sugar in grammes per litre, the experiment being performed on a liquid clarified with 10 per cent. of basic acetate of lead in a 200-millimetre tube.

LXXXVI.—TABLE FOR USE IN THE SACCHARIMETRICAL EXAMINATION OF URINE.—100 C.C. OF URINE CLARIFIED WITH 10 C.C. OF BASIC ACETATE OF LEAD.

Degrees observed in a 200-Millimetre Tube.	Grammes of Sugar per Litre of Urine.	
	French Saccharimeter.	German Saccharimeter.
1	2·25	3·61
2	4·50	7·23
3	6·75	10·83
4	9·00	14·88
5	11·25	18·05
6	13·50	21·66
7	15·75	25·27
8	18·00	28·88
9	20·25	32·49
10	22·50	36·10
11	24·75	39·71
12	27·00	43·32
13	29·25	46·93
14	31·50	50·54
15	33·75	54·15

SACCHARIMETRICAL ESTIMATION OF THE MILK SUGAR PRESENT IN MILK

The percentage of milk sugar in milk may easily be determined optically. 25 c.c. of a concentrated solution of neutral acetate of lead are added to 25 c.c. of the milk to be tested, and the mixture boiled. After cooling, the bulk is made up to 100 c.c., filtered, and polarised

in a 200-millimetre tube. The degrees observed, multiplied by 0.4103 for French saccharimeters, or by 0.6564 for the German instrument, give the amount of sugar in 100 c.c. of the milk.

The precipitate of albumenoid substances in 50 c.c. of milk occupy about 2 c.c. The bulk is made up to 102 c.c., so as to have 100 c.c. of liquid. In default of a flask graduated to 102 c.c., the result is multiplied by 0.98.

The milk may likewise be clarified by the addition of 1 c.c. of nitrate of mercury, *i.e.* a solution of mercury in twice its weight of nitric acid.

Harvey and Wiley clarify the milk by a solution of mercuric iodide, prepared by mixing—

	Grammes.
Potassium iodide (KI)	32.2
Mercuric chloride (HgCl ₂)	13.50
	Cubic centimetres.
Glacial acetic acid	25.00
Distilled water	640.00

Solution may be effected either in the hot or the cold. 50 c.c. of milk give an immediate precipitate with 25 c.c. of this solution. Water is added to the 100 c.c. mark, shaken, filtered, and polarised in the manner described above.

THE UNITED STATES OFFICIAL METHOD

The estimation of sugar.—The reagents, apparatus, and manipulation necessary to give the most reliable results in milk sugar estimation are as follows :—

Reagents.—(1) *Basic plumbic acetate*, specific gravity 1.97. Boil a saturated solution of sugar of lead with an excess of litharge, and make it of the strength indicated above. 1 c.c. of this will precipitate the albumens in 50 to 60 c.c. of milk.

(2) *Acid mercuric nitrate.*—Dissolve mercury in double its weight of nitric acid, specific gravity 1.42. Add to the solution an equal volume of water. 1 c.c. of this reagent is sufficient for the quantity of milk mentioned above. Larger quantities can be used without affecting the results of polarisation.

(3) *Mercuric iodide with acetic acid.*—KI, 33.2 grammes ; HgCl₂, 13.5 grammes ; C₂H₄O₂, 20 c.c. ; H₂O, 64 c.c.

Apparatus.—(1) Pipettes marked at 59.5, 60, and 60.5 c.c. (2) Sugar flasks marked at 102.4 c.c. (3) Filters, observation tubes, and polariscope. (4) Specific gravity spindle and cylinder. (5) Thermometers.

Manipulation.—(1) The room and milk should be kept at a constant temperature. It is not important that the temperature should be of any given degree. The work can be carried on equally well at

15° C., 20° C., or 25° C. The slight variations in rotatory power within the above limits will not affect the result for analytical purposes. The temperature selected should be the one which is most easily kept constant.

(2) The specific gravity of the milk is determined. For general work this is done by a delicate specific gravity spindle. Where greater accuracy is required, use specific gravity flasks.

(3) If the specific gravity be 1·026, or nearly so, measure out 60·5 c.c. into the sugar flask. Add 1 c.c. of mercuric nitrate solution, or 30 c.c. of mercuric iodide solution, and fill to the 102·4 c.c. mark. The precipitated albumen occupies a volume of about 2·44 c.c. Hence the milk solution is really 100 c.c. If the specific gravity is 1·030, use 60 c.c. of milk. If the specific gravity is 1·034, use 59·5 c.c. of milk.

(4) Fill up to the mark in the 102·4 c.c. flask, shake well, filter, and polarise.

NOTES.—In the above method of analysis the specific rotatory power of milk sugar is taken at 52·5, and the weight of it in 100 c.c. solution to read 100 degrees in the cane-sugar scale at 20·56 grammes. This is for instruments requiring 16·19 grammes sucrose to produce a rotation of 100 sugar degrees. It will be easy to calculate the number for milk sugar, whatever instrument is employed.

Since the quantity of milk taken is three times 20·56 grammes, the polariscopic readings divided by three give at once the percentage of milk sugar when a 200-millimetre tube is used.

If a 400-millimetre tube is employed, divide the reading by six; if a 500-millimetre tube is used, divide by 7·5.

Since it requires but little more time, it is advisable to make the analysis in duplicate, and take four readings for each tube. By following this method, gross errors of observation are detected and avoided.

By using a flask graduated at 102·4 for 60 c.c., no correction for volume of precipitated casein need be made. In no case is it necessary to heat the sample before polarising.

Alternate method.—The sugar may also be determined, either gravimetrically or volumetrically, by alkaline copper solution. (See Table XIV. pp. 45, 46.)

HONEY

The polarimetric examination of honey is intended to ascertain whether it be sophisticated with cane sugar or starch syrup. Biesterfeld recommends the following process:—

A 20 per cent. solution of honey is prepared, decolorised if need be with animal charcoal, and divided into two parts. The one is polarised as it stands, whilst the other is heated for ten minutes with

a few drops of sulphuric acid, cooled, brought to the original bulk, and polarised. If the honey be pure, Solution I. will have a levo-rotary power of 5° , and Solution II. will also be levo-rotary.

If Solution I. deviates to the right and Solution II. to the left, then the honey is sophisticated with cane sugar; if the two solutions deviate to the right, glucose is the adulterating agent; whilst, if Solution II., although still being dextro-rotary, deviates much less than Solution I., it may be concluded that the honey is sophisticated with both cane sugar and glucose.

The honey is also titrated with Fehling's solution (p. 13). It ought to contain at least 62 per cent. of reducing sugar.

ANALYSIS OF JAM—DETECTION OF GLUCOSE

1. Twenty grammes are dissolved in 200 c.c. of water and the solution halved. One half is inverted by a few drops of sulphuric acid, and the cane sugar and invert sugar estimated by Fehling's solution.

2. Five c.c. of basic acetate of lead are added to 50 c.c. of Solution I., filtered and polarised. After inversion the deviation should be to the left. From this deviation the amount of invert sugar is calculated. It should fairly correspond with that found with Fehling's solution.

3. If after inversion the liquid deviates to the right, the jam contains glucose, the presence of which is confirmed as follows: 200 grammes of the jam are dissolved in a litre of water and fermented as long as CO_2 is evolved, the liquid is then dialysed, renewing the dialysed liquid daily until it shows no further deviation, *i.e.* until it contains no more sugar. The liquid remaining is decolorised by bone black and polarised. If a deviation to the right be observed, and the liquid when tested by Fehling's solution shows no glucose, and the dextro-rotary substances are insoluble in absolute alcohol, the original *jam* contains glucose, as this reaction is characteristic of the accessory amylaceous products present in commercial glucose.

INDEX

- ACETATE of lead, corrections for, 63.
 Acetic acid, action of, on sugar, 5.
 Acids, action of, on glucose, 8, 39.
 " action of, on lactose, 14.
 " action of, on sugar, 4, 5.
 Air-pump for vacuum pan, 359.
 Air-pumps for condensers, 221, 223.
 Alcohol, action of, on maltose, 12.
 " phenylic, 9.
 " pulp-testing with, 63.
 L Alcoholic separation method, 270.
 Almonds, oil of, 10.
 Ammoniacal pumps, 230.
 Amygdalin, 10.
 Analysing apparatus, 16, 18.
 Analysis, Clerget's method, 41.
 " of expressed juice, 54, 61, 120.
 " of glucose, 394.
 " of limestone, 143.
 " of molasses, 383.
 " of raw sugar, 390.
 " of sugar, 14, 26, 332.
 " Pellet's continuous, 60.
 Ash, estimation of, 392, 393.
 Automatic diffusion process, 112.
 " register, Horsin-Deon's, 132, 133.
 " register, Langen's, 135, 136.
 " sampling of syrup, 234.
 Bag filters, Puvrez's, 171.
 " Taylor's, 339.
 Bagasse fuel, 299.
 " furnace, 300.
 " washing, 297, 298.
 Barium hydrate, carbonatation with, 130.
 Baryta, carbonatation with, 130, 131.
 " solubility of, 131.
 " testing sugar with, 9.
 Bases, action of, on glucose, 9.
 Beanes' charcoal process, 355.
 Beet-juice analysis, 55, 61, 64, 120.
 " automatic sampling of, 133.
 " chemical estimation of, 66.
 " density of, 50, 52, 120.
 " extraction by pressure, 76.
 " hydrometric testing of, 52, 120.
 " liming, 126.
 " measuring tanks for, 131, 132.
 Beet pulp, preparing, 56.
 Beet slices, size of, 85, 86.
 Beet sludge, composition of, 158.
 Beet sugar, cost of producing, 280.
 " molasses, 264.
 " preparing by diffusion, 78, 81, 96.
 Beets, chemical composition of, 119.
 " contract purchase of, 48, 50.
 " double-carbonatation process for, 78, 126.
 " drying machines for, 71, 72.
 " elevators for, 73.
 " heading and tailing, 49.
 " macerating, 77.
 " sampling of, 48, 49, 51.
 " slicing machinery for, 85, 90.
 " stone eliminators for, 69, 70, 93.
 " transporting, 67.
 " washing, 68, 69.
 " weighing, 71, 73.
 Beetroot pulp sampler, 95.
 Benzoic acid, incineration with, 393.
 Bessemer's expressing machine, 295.
 Bioses, composition of, 1, 2, 6.
 " crystallisation of, 3.
 " decomposition of, 2.
 " fermentation of, 2, 4.
 Bleaching process, Scoffern's, 344.

- Bleaching sugar, 162, 256, 337, 341, 344, 378.
 „ with ozone, 345.
 „ with sulphurous acid, 345.
 Blending sugar, 333.
 Boiling by doubles, 371.
 „ by thread-proof, 371.
 „ point in condensers, 209.
 „ sugar, 356, 366, 371.
 Bone char, analysis of, 163, 343.
 „ bleaching by, 162, 341, 346.
 „ cleaning by Beanes' process, 355.
 „ production of, 162.
 „ purifying by, 161, 321.
 „ revivifying, 161, 167, 169, 321, 348.
- CALCIMETRIC apparatus, 148.
 Calorie, definition of, 187.
 Calorisators, 81, 84, 100, 127, 307.
 Cambray's purification process, 325.
 Cane chips as fuel, 302.
 Cane juice, claying, 329.
 „ concentrating, 329.
 „ defecating, 324.
 „ dilution of, 316.
 „ evaporation of, 328.
 „ fermentation of, 323.
 „ impurities in, 325.
 „ neutralising, 324.
 „ properties of, 323.
 „ sulphitation of, 326, 327.
 Cane rollers, speed of, 294.
 Cane shredders, 291, 296, 306.
 Cane-sugar machinery, 291, 302, 306, 330, 374.
 „ properties, 3, 26.
 „ rotary power of, 12, 23, 390.
 „ solubility of, 3, 4.
 „ sources of, 1.
 „ taste of, 3.
 Cane-works, sampling in, 320.
 „ yield from, 321.
 Caramel, production of, 5.
 Carbonatation by barium hydrate, 130.
 „ by baryta, 130.
 „ by soda crystals, 130.
 „ continuous, 138.
 „ heat radiators in, 140, 141.
 „ process, 118.
 „ tanks, 136, 137.
 Carbonated juice, purity of, 150.
 Carbonic-acid injections, 126.
 Carbohydrates, 1.
- Cerosin, 284.
 Charcoal, old, 161, 167, 321, 351, 353.
 „ filters for juice, 159, 160, 164.
 „ vegetable, 342, 344.
 Chip chute, 308.
 Chlorine, action of, on syrup, 5.
 Chylaricose, 41.
 Circular diffusion batteries, 98, 307.
 Clarification during diffusion, 311, 313.
 Classen's experiments, 210.
 Claying cane juice, 329.
 „ sugar loaves, 378.
 Clerget's formula, 384.
 „ method of analysis, 41.
 Colloids, 80.
 Concentrating cane juice, 329.
 Condense box, 365.
 Condensation vacuum, 188.
 Condenser, injection, 360.
 Condensers, air-pumps for, 221, 223.
 „ barometrical, 222.
 „ capacity of, 366.
 „ pressure in, 223.
 Coniferin, 9.
 Continuous carbonatation, 138.
 Cooler, Bocquin-Lepchinsky's, 243, 255, 274.
 „ Ragot and Tournour's, 279.
 Copper alkali solution tests, 32.
 Copper solution tests, 26, 32, 34, 38.
 Corrections for acetate of lead, 63.
 Counter-current condensers, 223.
 Creydt's formula, 386.
 Crystallisation in motion, 243, 263, 273.
 „ of sugar, 2, 3, 367.
 Crystalloids, 80, 81.
 Cycle diffusion, 84.
- DECOMPOSITION of bioses, 2.
 „ of monoses, 2.
 Defecation by lime, 117, 121, 123, 146, 248.
 „ by sulphuric acid, 118.
 „ of cane sugar, 345.
 Definition of sugars, 1.
 Dehne's extraction system, 153, 156.
 Demerara sugar, 329.
 Density of beet juice at different temperatures, 50, 120.
 „ of cane-sugar solutions, 54.
 Dextrose, composition of, 2, 40.
 Dextrine, composition of, 11.
 Diabetes, glucose in, 6, 7, 33, 398.
 Dialysis, 79, 80, 83, 266.
 Diastase, 11.

- Diffuser, compressed-air, 85.
 Diffusers, charging, 98, 99.
 Diffusion and pressed juice compared, 129.
 ,, batteries, temperature in, 314, 315.
 ,, juice, quality of, 83.
 ,, machinery, 96, 108.
 ,, of sugar-cane, 301, 306, 307, 311, 316, 317.
 ,, process, 78, 81, 96.
 ,, process, automatic, 112.
 ,, records, 308, 309, 310.
 ,, residuals, 103, 106.
 Dilution of cane juice, 316.
 Distillation of sugar, 6.
 ,, process of, 177.
 Double-carbonatation process, 78, 126, 345.
 Drying machines for beets, 71, 72.
 ,, sugar loaves, 379.
 Dulcitol alcohol, 14.
- EFFECTIVE heat, 203.
 Elevators for beets, 73.
 Encrustation in condensers, 208.
 Esculin, 9.
 Estimation tables, 38, 43-47.
 Evaporation by steam, 180, 187.
 ,, of cane juice, 328.
 ,, open pan, 179, 181.
 ,, process of, 177.
 Exhausted pulp, 103, 106, 110.
 ,, analysis of, 110, 111.
 ,, presses for, 104, 106.
 Expressing machine, Bessemer's, 295.
- FEHLING'S solution, 13, 26, 28, 32, 42, 384, 391, 394, 402.
 Fermentation of bioses, 2, 4.
 ,, of maltose, 12.
 ,, of monoses, 2.
 Filter-press pumps, 155.
 Filter-presses, 125, 152, 170, 341.
 Filter-tanks, 346.
 Filters, 152-154, 170, 339.
 ,, charcoal, for juice, 159, 160.
 ,, mechanical, for juice, 162, 170.
 Filtration of limed juice, 147.
 ,, plant in France, 176.
 "Flash," the, 368.
 Froth curbs, 137.
 Frothing, prevention of, 137, 193, 215.
- GAS pump for sugar-works, 145.
 Gas purifiers for sugar-works, 144, 145.
- Glucose, action of acids on, 8.
 ,, action of bases on, 9.
 ,, action of heat on, 8.
 ,, analysis of, 394.
 ,, composition of, 1, 2, 6.
 ,, effect of, on crystallisation, 371.
 ,, estimation of, 28, 31, 35, 394.
 ,, in honey, 7.
 ,, in jam, 402.
 ,, production of, 7.
 ,, properties of, 8.
 ,, solubility of, 8.
 ,, titrating with copper solution, 30.
- Glucosic ethers, 9.
 Glucosides, 9.
 Grape sugar, sources of, 1, 6.
 Gravimetric analysis of sugar, 42-47, 391.
 Green syrup, 377, 386.
- HEAT, action of, on glucose, 8.
 ,, ,, on levulose, 11.
 ,, ,, on sugar, 5, 6.
 ,, ,, on sugar syrups, 190.
 ,, coefficients of transmission of, 199.
 ,, effective, 203.
 ,, radiators in carbonatation, 140, 141.
 ,, radiators in evaporating, 219.
 Heating surface, calculation of, 211.
 Hexelose, 40.
 "Hodek" steam sifter, 217.
 Honey, testing, 401.
 Howard's vacuum pan, 180, 187, 188, 190, 357.
 Hughes' cane cutter, 306.
 Hydration products of starch, 12.
 Hydrochloric acid, action of, on levulose, 39.
 ,, ,, action of, on glucose, 39.
 ,, ,, washing charcoal with, 355.
- INJECTION piping in tanks, 137.
 ,, condenser, 360.
 Inulin, 10.
 Inversion experiments, 39.
 ,, process of, 40.
 Invert sugar, 4, 6, 10, 26, 40.
 ,, estimations, 37, 38.
- JAM testing, 402.
 Jelinek's evaporation calculations, 202.

- Joint making, 363.
 Juice concentration tables, 184.
- KONIG'S sugar recoverer, 217, 218.
- LABORATORY, arrangement of, 16.
 Lactose, action of acids on, 14.
 ,, production of, 13.
 ,, properties of, 13.
 ,, rotary power of, 13.
 ,, sources of, 1.
 Latent heat of steam, 187.
 Levulose, action of heat on, 11.
 ,, composition of, 2, 10, 40.
 ,, production of, 10, 11.
 ,, rotary power of, 11.
 ,, solubility of, 11.
 Levulosane, composition of, 5.
 Lime, deficiency of, 248.
 ,, excess of, 248.
 ,, milk of, preparing, 146.
 ,, percentage in animal charcoal, 166.
 ,, use of, in diffusion, 313, 314.
 ,, use of, in refining, 337, 352.
 Lime kilns for sugar-works, 142, 144.
 Limed juice, titration of, 147.
 Limestone, analysis of, 143.
 Line diffusion batteries, 97, 307.
 Liquoring sugar loaves, 378.
 Loaf-sugar bleaching, 162.
- MACERATING beets, 77.
 Mactose, 40.
 Maltose, action of alcohol on, 12.
 ,, fermentation of, 12.
 ,, production of, 11.
 Mannite alcohol, 14.
 Manoury's separation method, 271.
Masse cuite, 240, 243-245, 258, 260, 327.
 Measuring tanks for beet juice, 131, 132.
 Mechanical filter, Daneck's, 172.
 ,, Phillippe's, 173.
 Melitrose, properties of, 386.
 Milk sugar, estimation of, 399.
 ,, sources of, 1.
 Mixing sugar, 257.
 Molasses, analysis of, 383.
 ,, beet sugar, 264, 265.
 ,, cane sugar, 329.
 ,, chemistry of, 382.
 ,, density of, 383.
 ,, elimination of, 273, 275.
 ,, treatment of, 265, 266.
 ,, use of, 381.
 Monoses, composition of, 1, 2, 6, 40.
 Monoses, crystallisation of, 3.
 ,, decomposition of, 2.
 ,, fermentation of, 2.
 Montejus, use of, 155, 336, 338.
 Moulds, sugar-loaf, 376.
 Multiple-effect calculations, 200, 202, 205, 209.
 ,, system, economy of, 228.
 ,, vacuum plant, 180, 191, 196, 198.
- NICOL prisms, 21.
 Nitric acid, action of, on sugar, 5.
 ,, ,, on glucose, 8, 39.
- OPEN-PAN evaporation, 179, 181.
 Osmogènes, 266, 267, 268.
 Osmosis, 79, 80, 83, 266.
 Ozone, bleaching with, 345.
- PECTIC fermentation of beet juice, 121.
 Pectose fermentation of beet juice, 121.
 Perichon's washing process, 297.
 Phenyl alcohol, 9.
 Polariscopes, 15, 19, 22.
 Polariscopes, temperature corrections for, 22.
 ,, use of, 14, 16, 18, 21.
 Presses for exhausted pulp, 104, 106.
 Pressure, beet juice extraction by, 76.
 Proof, examination of, 369.
 Proof-stick, cleansing the, 372.
 Proof-sticks, 359, 368.
 Pulp, eliminating machine, 114, 115.
 ,, exhausted, 103, 106.
 ,, pressing after diffusion, 103.
 ,, testing, 59.
 Pulping apparatus, 57, 58.
 Purification process, Cambray's, 325.
 Puvrez's sulphitation method, 272, 273.
- QUADRUPLE-EFFECT apparatus, 226.
 Quintuple-effect apparatus, 226.
- RADIATING heater, Witkowicz's, 219.
 Raffinose, estimation of, 387.
 ,, properties of, 386.
 Ralentisseur in condensers, 217.
 Raw sugar, analysis of, 390.
 Reagents of sugars, 26, 34.
 Refining sugar, 331, 333, 336, 356.
 Register, Langen's automatic, 135, 136.
 ,, Horsin-Deon's automatic, 132, 133.
 Reheating apparatus, 230, 373.
 Revivifying bone char, 348.

- Rillieux's inventions, 182, 192, 194, 233, 241.
 Rotary power of cane sugar, 12, 23, 384, 390.
 " " of glucose, 5.
 " " of lactose, 13.
 " " of levulose, 5, 11, 41.
 " " of maltose, 11, 12.
 SACCHARIMETER, portable, 21.
 " " use of, 383, 390, 399.
 Saccharimeters, optical, 14, 17, 21.
 Saccharimetrical valuation, 52, 53.
 Saccharide, composition of, 5.
 Saccharose, action of acetic acid on, 42.
 Saccharoses, composition of, 1, 2, 9.
 Safety vessel in condensers, 215, 217.
 Salicin, 9.
 Salizenin, 9.
 Sampling of beet juice, automatic, 133.
 " " in cane-works, 320.
 " " syrup when boiling, 358, 368.
 Sangerhausen cane-cutter, 304, 305.
 Scheller's estimation reagent, 37.
 Scoffer's bleaching process, 344.
 "Seconds," use of, 258, 277, 334.
 Separation method, Strontia, 271.
 " " Manoury's, 271.
 " " Steffen's, 268, 272, 274, 381.
 Siloing pressed pulp, 106.
 Slicing machinery for beets, 85, 90.
 " " knives for, 87.
 " " speed of, 87, 94.
 Sludge-cake manure, 157.
 Sludge mashing, 157.
 " " washing, 155.
 Soda crystals, carbonatation with, 130.
 Soldaini's reagent, 35.
 Solubility of cane sugar, 3, 4.
 " " of glucose, 8.
 " " of levulose, 11.
 " " of sugar, 236.
 Solutions, heating of, 335.
 Soxhlet's methods of analysis, 37, 42, 397.
 Standard solution of sugar, 26.
 Starch, hydration products of, 12.
 Steam calculations, 187.
 " " consumption table, 259.
 " " regulation for pan, 364.
 Steffen's separation method, 268, 272, 274, 381.
 Stoektaking, 246.
 Stone eliminators for beets, 69, 70, 93.
 Stone eliminators for pumps, 150, 151.
 Strontia, separation method, 271.
 " " solubility of, 131.
 Sucrate of lime, separation process, 268.
 Sucrates of lime, 122.
 Sucrose, sources of, 1.
 Suero-carbonates, 122, 123.
 Sugar, action of acids on, 4, 5.
 " " analysis of, 14, 26, 332.
 " " bleaching, 256, 257, 337, 341, 344, 378.
 " " blending, 333.
 " " boiling, 356, 366.
 " " hindrances to, 247.
 " " "in grain," 240.
 " " point of, 238.
 " " "to proof," 240.
 " " brown, 260.
 " " cane, analysing the, 289.
 " " " composition of, 282, 283.
 " " " cost of, 286, 287, 288.
 " " " culture, 284, 285.
 " " " cutters, 304, 305, 306.
 " " " diffusion, 301, 306, 307, 311, 316, 317.
 " " " mills, 290, 291, 293.
 " " " reproduction of, 284.
 " " crystallisation of, 2, 3, 367.
 " " distillation of, 6.
 " " fermentation of, 2, 4.
 " " gravimetric analysis of, 42-47, 391.
 " " invert, 4, 6, 10, 26, 40.
 " " loaf moulds, 376.
 " " mixing, 257.
 " " purity of, 261, 262, 263.
 " " qualities of, 258, 260.
 " " reactions of, 2, 4, 26.
 " " recoverer, Konig's, 217, 218.
 " " refining, 331, 333, 336, 356.
 " " solubility of, 236.
 " " solutions, boiling-points of, 182.
 " " standard solution of, 26.
 " " tablets, 386.
 " " total estimation of, 27, 29.
 " " uncrystallisable, 391.
 " " volumetric analysis of, 26, 27, 29, 31, 390.
 " " warehouses, 264.
 " " works, gas pump for, 145.
 " " " gas purifiers for, 144, 145.
 " " " lime kilns for, 142, 144.
 Sugars, chemical composition of, 1, 6.
 " " classification of, 1, 257, 331.
 " " definition of, 1.

- Sulphitation method, Quarez's, 272, 273.
 ,, of cane juice, 326, 327.
 ,, pans, 327.
- Sulphuric acid, action of, on sugar, 5.
 ,, incineration, 392.
- Sulphurous acid, bleaching with, 345.
- Syrups, composition of, 388.
- TABLET sugar, 386.
- Tanks, carbonatation, 136, 137.
 ,, Schutzenback's, 244, 246, 255.
- Taylor's bag filters, 339.
- Temperature, fall of, in condensing, 212.
 ,, in diffusion batteries, 314, 315.
- Thread-proof, boiling by, 371.
- Triple-effect apparatus, 213, 220, 232.
- Tube fixing, Austrian method, 233.
- Turbine speeds, 251.
 ,, statistics, 250.
 ,, Weston's, 252.
- Turbining sugar, 249, 334.
 ,, sugar tablets, 380.
- Turbines, centrifugal force of, 249.
 ,, feeding, 254, 256.
 ,, types of, 251.
- UNCRYSTALLISABLE sugar, 391.
- VACUUM pan, Howard's, 357.
 ,, ,, testing, 362, 365.
 ,, ,, use of, 361, 366.
 ,, pans, feeding, 369.
 ,, ,, Lexa's, 241.
 ,, ,, vertical, 238, 239.
 ,, plant, multiple-effect, 180, 181, 191, 196, 198.
- Vaporisation statistics, 183, 186.
- Vats, cooling, 243.
- Violette's analysing process, 65.
- Volumetric analysis of sugar, 26, 27, 29, 31, 390.
- WASHING machines for beets, 68, 70, 72.
 ,, sugar loaves, 379.
- Weighing machines for beets, 71, 73, 74, 75.
- Wiley's analysing process, 394.
- Wines, testing, 396.
- Witkowicz's radiating heater, 219.
- YARYAN apparatus, 234, 311.
- ZINC oxide, estimation by, 393.

EVAPORATING, CONDENSING,

AND

COOLING APPARATUS.

Explanations, Formulæ, and Tables for Use in Practice.

By **E. HAUSBRAND, Engineer.**

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By **A. C. WRIGHT, M.A. (Oxon.), B.Sc. (Lond.)**

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TABLE OF CONTENTS.

Chapters I. The Coefficient of Transmission of Heat, h , and the Mean Temperature Difference, θ_m .—II. Parallel and Opposite Currents.—III. Apparatus for Heating with Direct Fire.—IV. The Injection of Saturated Steam.—V. Superheated Steam.—VI. Evaporation by Means of Hot Liquids.—VII. The Transference of Heat in General, and Transference by Means of Saturated Steam in Particular.—VIII. The Transference of Heat from Saturated Steam in Pipes (Coils) and Double Bottoms.—IX. Evaporation in a Vacuum. X. The Multiple-effect Evaporator. A. *The Evaporative Capacity of Each Vessel.* B. *The Percentage of Dry Material in the Liquid in Each Vessel.*—XI. Multiple-effect Evaporators, from which Extra Steam is taken.—XII. The Weight of Water which must be Evaporated from 100 Kilos. of Liquor in order to bring its Original Percentage of Solids from 1.25 per cent. up to 20.70 per cent.—XIII. The Relative Proportion of the Heating Surfaces in the Elements of the Multiple Evaporator and their Real Dimensions.—XIV. The Pressure Exerted by Currents of Steam and Air upon Floating Drops of Water.—XV. The Motion of Floating Drops of Water, upon which press Currents of Steam.—XVI.

The Splashing of Evaporating Liquids.—XVII. The Diameter of Pipes for Steam, Alcohol Vapour, and Air.—XVIII. The Diameter of Water Pipes.—XIX. The Loss of Heat from Apparatus and Pipes to the Surrounding Air, and Means for Preventing the Escape.—XX. Condensers. A. *Jet Condensers.* B. *Surface Condensers (Coolers).*—XXI. Heating Liquids by Means of Steam. A. *Steam Heating Coils in the Liquid.* B. *Steam Vessels with Double Bottoms.* C. *The Liquid flows through Tubes around which Steam is at rest.*—XXII. The Cooling of Liquids. A. *The Direct Introduction of Ice.* B. *The Direct Addition of Cold to Hot Liquid.* C. *By Partial Evaporation.* D. *By Means of a Colder Liquid.* E. *Open Surface-Coolers.* F. *By Contact with Metallic Surfaces Cooled by Air.* G. *Direct Cooling by Means of Air.* H. *Cooling Air by Means of Water.*—XXIII. The Volumes to be Exhausted from Condensers by the Air-Pumps.—XXIV. A Few Remarks on Air-Pumps and the Vacua they Produce.—XXV. The Volumetric Efficiency of Air-Pumps.—XXVI. The Volumes of Air which must be Exhausted from a Vessel in order to Reduce its Original Pressure to a Certain Lower Pressure.—INDEX.

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INDEX TO SUBJECTS.

PAGE		PAGE		PAGE
10	Agricultural Chemistry ...	30	Dyeing Marble ...	6
11	Air, Industrial Use of ...	22	Dyeing Woollen Fabrics ...	2
9	Alum and its Sulphates ...	23	Dyers' Materials ...	27
9	Ammonia ...	23	Dye-stuffs ...	18
3	Aniline Colours ...	19, 20	Enamelling Metal ...	14
6	Animal Fats ...	18	Enamels ...	16
4	Anti-corrosive Paints ...	31	Engraving ...	20
30	Architecture, Terms in ...	7	Essential Oils ...	30
16	Architectural Pottery ...	26	Evaporating Apparatus ...	3
7	Artificial Perfumes ...	27	External Plumbing ...	3
10	Balsams ...	5, 6	Fats ...	10
23	Bleaching ...	21	Faults in Woollen Goods ...	12
8	Bone Products ...	26	Gas Firing ...	16
31	Bookbinding ...	17	Glass-making Recipes ...	9
15, 16	Brick-making ...	18	Glass Painting ...	11
27	Burnishing Brass ...	8	Glue Making and Testing ...	23
21	Carpet Yarn Printing ...	5	Greases ...	19
14, 15	Ceramic Books ...	17	History of Staffs Potteries ...	25
8	Charcoal ...	28	Hops ...	7
9	Chemical Essays ...	28	Hot-water Supply ...	20
17	Chemistry of Pottery ...	13	India-rubber ...	20
23	Chemistry of Dye-stuffs ...	3, 11	Inks ...	11
16	Clay Analysis ...	4	Iron-corrosion ...	31
26	Coal-dust Firing ...	26	Iron, Science of ...	26
21	Colour Matching ...	28	Japanning ...	30
25	Colliery Recovery Work ...	27	Lacquering ...	16
21	Colour-mixing for Dyers ...	3	Lake Pigments ...	4
15	Colouring Pottery ...	11	Lead and its Compounds ...	20
22	Colour Theory ...	13	Leather Industry ...	20
24	Combing Machines ...	14	Leather-working Materials ...	19, 20
6	Compounding Oils ...	31	Lithography ...	29
26	Condensing Apparatus ...	5, 6	Lubricants ...	4
7	Cosmetics ...	8, 10	Manures ...	7
22	Cotton Dyeing ...	2	Mineral Pigments ...	10
24	Cotton Spinning ...	25	Mine Ventilation ...	12
20	Damask Weaving ...	25	Mine Haulage ...	32
29	Dampness in Buildings ...	3	Oil and Colour Recipes ...	32
28	Decorators' Books ...	4	Oil Boiling ...	29
20	Decorative Textiles ...	5	Oils ...	30
27	Dental Metallurgy ...	12	Ozone, Industrial Use of ...	22
3	Dictionary of Paint Ma- terials ...	2	Paint Manufacture ...	11
5	Drying Oils ...	3	Paint Materials ...	11
11	Drying with Air ...	4	Paint-material Testing ...	13
		18	Paper-pulp Dyeing ...	20
			Petroleum ...	
			Pigments, Chemistry of ...	
			Plumbers' Work ...	
			Porcelain Painting ...	
			Pottery Clays ...	
			Pottery Manufacture ...	
			Power-loom Weaving ...	
			Preserved Foods ...	
			Printing Inks ...	
			Recipes for Oilmen, etc. ...	
			Resins ...	
			Risks of Occupations ...	
			Rivetting China, etc. ...	
			Scheele's Essays ...	
			Sealing Waxes ...	
			Silk Dyeing ...	
			Silk Throwing ...	
			Smoke Prevention ...	
			Soaps ...	
			Spinning ...	
			Staining Marble, and Bone ...	
			Steam Drying ...	
			Sugar Refining ...	
			Steel Hardening ...	
			Sweetmeats ...	
			Terra-cotta ...	
			Testing Paint Materials ...	
			Testing Yarns ...	
			Textile Fabrics ...	
			Textile Materials ...	
			Timber ...	
			Varnishes ...	
			Vegetable Fats ...	
			Waste Utilisation ...	
			Water, Industrial Use ...	
			Water-proofing Fabrics ...	
			Weaving Calculations ...	
			Wood Waste Utilisation ...	
			Wood Dyeing ...	
			Wool Dyeing ...	
			Writing Inks ...	
			X-Ray Work ...	
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