PRACTICAL CEMENT TESTING

BY

W. PURVES TAYLOR, M.S., C.E.

Engineer in Charge Philadelphia Municipal Testing Laboratories

NEW YORK
MYRON C. CLARK
13-21 Park Row
1905

PREFACE.

Although during the past year or two, the additions to the literature on cement and concrete have been especially notable, it nevertheless has appeared to the author that a complete description of the methods of handling practical tests of cement has not yet been brought before the public. The methods usually given in text books assume too much knowledge for the use of the student or beginner, while to the practical operator the directions are too general to be of much value. The excellent standard methods of testing recently issued by committees of several technical societies have already done much to promote uniformity in testing, but are of little real assistance either to the novice or the expert in enabling him to increase the accuracy or to simplify the routine of his work. This volume therefore has been designed primarily for the use of the student, the novice, and the practical operator in conducting actual routine tests of cement to determine its suitability for purposes of construction, but it is hoped that both the expert and the engineer who directs this work may also find something of interest in its pages.

The general scope of the book covers a description of the properties of cement, the objects of the various tests, the methods of conducting them, the common influences and errors that are most likely to affect the determinations, and the practical interpretation of the results which are finally obtained. No attempt has been made to consider the practical use of cement and concrete except in so far as the conditions of actual work regulate the use of the various tests, while the data given are also applicable more to the conduct of tests than to the final use of the material. In other words, the scope of the book is intended to cover only the methods and the application of the tests of cement commonly employed in routine work, and not to consider theoretical properties, investigations of a research character, nor the use of cement.

Chapters I., II. and III. are of an introductory nature, and are included only for the information of the student, and for the logical development of the subject. The constitution of cement is considered very briefly, while the chapter on manufacture is purely descriptive and makes no attempt at being technical.

The body of the book, Chapters IV. to XII., is devoted to the conduct of routine tests of cement. The chapter on Chemical Analysis was prepared in collaboration with Mr. Charles S. Reeve, Chemist of the Philadelphia Laboratories.

Chapter XIII. deals with simple tests by which the character of a cement may be ascertained with practically no apparatus, and which might be of considerable service to the small consumer or to the expert when it is impracticable to obtain apparatus to make tests in the orthodox manner.

The practical operation of a cement laboratory is considered in Chapter XIV., which also describes the organization, the labor required, and the cost of testing.

To avoid an endless number of qualified statements, in describing the different properties and tests, the body of the book is devoted entirely to Portland cement, while in Chapter XV. is given a description of the properties and tests of natural, improved, slag, sand, and other varieties of cement.

Chapter XVI. is devoted to the subject of cement specifications, while the different Appendices give the standard specifications and methods of testing issued by various scientific bodies.

The methods of testing used in the author's laboratory are given at some length throughout the course of the book, and it is hoped that the frequent allusions to them will be pardoned on account of the long and varied experience of the laboratory, and on account of the large amount of testing performed, which has required both development of system and simplification of method.

The author wishes to express his appreciation of the kindness of Mr. George S. Webster, Chief Engineer of the City of Philadelphia, in permitting him to describe the methods employed, and to use data obtained in the Philadelphia Laboratories, and also to acknowledge the interest that has enabled the Laboratories to attain their present stage of development.

Acknowledgements for the use of cuts are also due to The Allis-Chalmers Co., J. W. Bramwell, The Bonnot Co., The Brad-

ley Pulverizer Co., The Cement Age, F. L. Smidth & Co., The Geo. V. Cresson Co., F. D. Cummer & Son Co., The Fairbanks Co., The Kent Mill Co., Lathbury & Spackman, Tinius Olsen & Co., Thos. Prosser & Son, Riehle Bros. Testing Machine Co., Frederick W. Taylor, Henry Troemner and the International Instrument Co.

W. PURVES TAYLOR.

January 1, 1906.

TABLE OF CONTENTS

PRACTICAL CEMENT TESTING

CHAPTER I.

CLASSIFICATION AND STATISTICS.

Definitions.—Hydraulic cement may be somewhat broadly defined as a material which, when pulverized and mixed with water into a paste, acquires the property of setting and hardening under water. In engineering construction, four classes of cement are generally recognized—(1) Portland cement, (2) natural cement, (3) Pozzuolana cement, (4) mixed or blended cement.

Portland cement is the product resulting from the process of grinding an intimate mixture of calcareous and argillaceous materials, calcining the mixture to incipient fusion, and grinding the resulting clinker to a fine powder. It must contain no materials added subsequent to calcination other than small amounts of certain substances used to regulate its setting properties. The German Association of Portland Cement Manufacturers has adopted the following definition of Portland cement:

"A hydraulic cementing material having a specific gravity of not less than 3.10 in the calcined condition, and containing not less than 1.7 parts by weight of lime to one part each of silica, alumina, and ferric oxide, the material being prepared by intimately grinding the raw ingredients, calcining them to not less than clinkering temperature, and then reducing this clinker to a proper fineness."

English and American societies* also have adopted standard definitions of Portland cement similar in tenor, although not as explicit, except that they limit the amount of substances that may be added after calcination to two or three per cent.

Natural cement is the product resulting from the burning and subsequent pulverization of an argillaceous limestone or other suitable rock in its natural condition, the heat of burning being insufficient to cause vitrification.

*See Appendices.

This class of cement is also commonly known as "Rosendale," it being so named from the district in the eastern part of New York State which is the greatest producer of natural cement. This term, however, when applied to all naturals is a misnomer. In England the name "Roman" is applied to certain grades of this material.

Pozzuolana cement is obtained by grinding together an intimate mixture of slaked lime and blast-furnace slag or volcanic scoria. The cement is not burned, the hydraulic ingredients being present only as a mechanical mixture. This material must not be confused with slag Portland, which is a regular Portland cement in which the slag furnishes the silicious ingredients, thus replacing the clay or shale in the mix.

Mixed cements cover a large variety of products made by combining the other forms of cement or by mixing them with an inert material. The so-called "second-grade" Portlands generally belong in this class, since they consist usually of Portland cement mixed either with natural cement or with raw rock, cinder or slag. Sometimes, however, these cements are merely made of inferior clinker, in which case they are to be classed with Portlands. "Improved cements" are naturals containing from 10 to 30 per cent. of Portland clinker. Sand cements are made by finely grinding a mixture of Portland cement and sand, usually in equal proportions. These varieties of mixed cements are those most commonly encountered, although many other forms are to be found on the market.

Distinguishing Characteristics.—The distinguishing characteristics of Portland cement are—in manufacture, the use of an artificial mixture, the grinding before burning, and the calcination to incipient fusion—and in use, its heavier weight, its slower set, and its greater strength.

Natural cement is distinguished in manufacture by its production from a single variety of material, unground, and burned at a low heat, and in use by its lighter weight, quicker set, and lower strength in the earlier stages of hardening.

In what follows in this book, the discussion will be limited to Portland cement (except for Chapter XV.), so that whenever the unqualified term **cement** *is employed, Portland cement alone is to be understood.*

Historical.*—Although Smeaton, when building the Eddystone lighthouse in 1756, discovered that the addition of clay to lime would render it capable of setting under water, no real Portland cement was produced until 1824, when Joseph Aspdin, a brick-layer, of Leeds, England, took out a patent for producing a cement by calcining a mixture of lime and clay. He gave to it the name "Portland" on account of its resemblance when hardened to the famous oölitic limestone, used extensively for building, found in the "Isle of Portland," a peninsula on the southern coast of England. The first works for producing this material were established at Wakefield by Aspdin in 1825, while the construction of the Thames tunnel in 1828 was the first important piece of engineering work to use it in any quantity.

This early cement, however, was very different from that of modern days, chiefly in that the burning was never carried up to the point of vitrification, so that the elements could never have been properly combined. It was not until about 1845 that the manufacture began to be placed upon a scientific basis, and Portland cement, as we now know it, to be produced. In Germany, the first works were established near Stettin in 1852, while 1875 marked the beginning of the industry in the United States.

Statistics of Industry.—The development of the Portland cement industry, particularly in the past decade, has been of such remarkable proportions, that a brief summary of statistics regarding it cannot fail to show the importance of the scientific study of a material so widely employed. The following data are taken largely from the report of the Geological Survey† for 1903:

Table I. shows the domestic production of Portland, natural and slag cements. It will be observed that the production of natural cement has remained practically constant for the past fifteen years, while the Portland production, particularly since 1895, is remarkable in its rapid increase. The chief reason that Portland cement production, prior to 1895, advanced so slowly, was the deep-seated prejudice of our engineers against

*For more complete data on the history of cement consult Redgrave's "Calcareous Cements," and Cummings' "American Cements."

†"The production of Cement in 1903," by L. L. Kimball—extract from "Mineral Resources of the United States" for 1903—issued by Division of Mining and Mineral Resources, U. S. Geological Survey.

the domestic and in favor of the foreign material. Even as late as the end of the past decade, it was not uncommon for the specifications of important work to call for foreign cement, or at least for a cement "that shall be equal to the best German brands." The excellence of the domestic product and its equality to the best foreign material is now, however, generally conceded.

TABLE I.—Total Production of Natural, Portland and Slag Cement in the United States, 1818-1903.

(From Mineral Resources of the United States, 1903.)

Year.	Natural.	Portland.	Pozzuolana or Slag.
	Number of Barrels.		
1818 to 1830	300,000	......	......
1830 to 1840	1,000,000	......	......
1840 to 1850	4,250,000	......	......
1850 to 1860	11,000,000	......	......
1860 to 1870	16,420,000	......	......
1870 to 1880	22,000,000	82,000	......
1880	2,030,000	42,000	......
1881	2,440,000	60,000	......
1882	3,165,000	85,000	......
1883	4,190,000	90,000	......
1884	4,000,000	100,000	......
1885	4,100,000	150,000	.....
1886	4,186,152	150,000	......
1887	6,692,744	250,000	......
1888	6,253,295	250,000	......
1889	6,531,876	300,000	......
1890	7,082,204	335,000	......
1891	7,451,535	454,813	......
1892	8,211,181	547,440	......
1893	7,411,815	590,652	......
1894	7,563,488	798,757	......
1895	7,741,077	990,324	......
1896	7,970,450	1,543,023	12,265
1897	8,311,688	2,677,775	48,329
1898	8,418,924	3,692,284	150,895
1899	9,868,179	5,652,266	335,000
1900	8,383,519	8,482,020	446,609
1901	7,084,823	12,711,225	272,689
1902	8,044,305	17,230,644	478,555
1903	7,030,271	22,342,973	525,896
Total	209,132,526	79,608,196	2,270,238

Table II. gives the distribution of production by States, Pennsylvania leading both in the number of mills and in quantity of output.

The distribution by sections (Table III.) divides the production into the "Lehigh district" of Pennsylvania and New Jersey, the New York district, both of which operate on limestone, and

TABLE II.—Production of Portland Cement in the United States in 1903, by States.

(From Mineral Resources of the United States, 1903.)

State.	Number of Works.	Quantity. Barrels.	Value.
Alabama	1	......	...
Arkansas	1	......	......
California	3	631,151	$1,019,352
Colorado	*a* 1	258,773	436,535
Georgia	2	......	......
Illinois	5	1,257,500	1,914.500
Indiana	3	1,077,137	1,347,797
Kansas	*b* 1	1,019,682	1,285,310
Michigan	13	1,955,183	2,674,780
Missouri	*c* 2	825,257	1 164,834
New Jersey	3	2,693,381	2,944,604
New York	12	1,602,946	2,031,310
Ohio	8	729 519	998,300
Pennsylvania	17	9,754,313	11,205 892
South Dakota	1	......	......
Texas	2	......	......
Utah	1	......	......
Virginia	*a* 1	538,131	690,105
West Virginia	1	......	......
Total	78	22,342,973	$27,713,319

(*a*) Includes product of Utah and South Dakota.
(*b*) Includes product of Texas.
(*c*) Includes product of Arkansas.
(*d*) Includes product of Alabama, Georgia and West Virginia.

TABLE III.—Showing the Development of the Portland Cement Industry in the United States.

(From Mineral Resources of the United States, 1903.)

Section.	1890. Number of works.	1890. Quantity. Barrels.	1890. Per cent.	1900. Number of works.	1900. Quantity. Barrels.	1900. Per cent.
New York	4	65,000	19 4	8	465,832	5.5
Lehigh and Northampton counties, Pa., and Warren County, N. J.	5	201,000	60.0	15	6,153,629	72.6
Ohio	2	22,000	6.5	6	534 215	6.3
Michigan	..	......	....	6	664,750	7 8
All other sections	5	47 500	14.1	15	663,594	7.8
Total	16	335,500	100.0	50	8,482,020	100.0

Section.	1902. Number of works.	1902. Quantity. Barrels.	1902. Per cent.	1903. Number of works.	1903. Quantity. Barrels.	1903. Per cent.
New York	10	1,156,807	6.8	12	1,602,946	7 2
Lehigh and Northampton counties, Pa., and Warren County, N. J.	17	10,829,922	62 8	16	12,324,922	55.2
Ohio	7	563,113	3.3	8	729,519	3 3
Michigan	10	1 577,006	9.1	13	1,955 183	8 7
All other sections	21	3,103,796	18 0	29	5,730,403	25 6
Total	65	17,230,644	100.0	78	22,342,973	100.0

the Ohio-Michigan district, which generally employs marl. The Lehigh district produces over half the Portland cement made in the country. The recent development of the industry in Michigan is also a feature of the table.

The diagram, Fig. 1, shows a comparison of the consumption

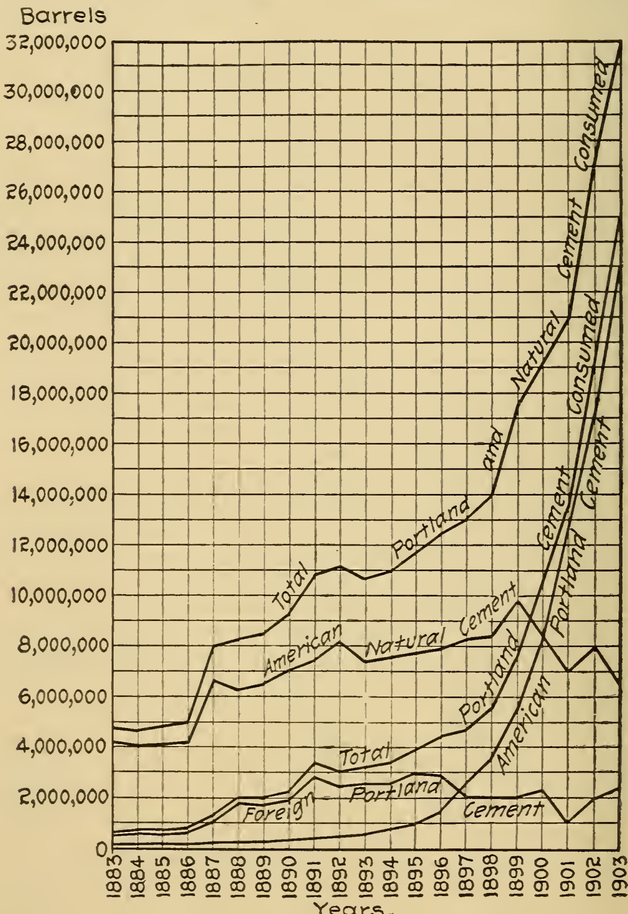


FIG. 1.—Diagram Showing the Quantity of Cement Consumed in the United States During the Twenty Years, 1883–1903.

of Portland cement in the United States with the entire amount of hydraulic cement imported in the last few years, which illustrates not only the growth of the industry, but also the marked increase in the ratio of domestic production to total consumption.

CHAPTER II.

COMPOSITION AND CONSTITUTION.

Composition.—The essential components of Portland cement are silica, alumina and lime. Other ingredients always occurring in appreciable quantities are iron, magnesia, alkalies, sulphuric and carbonic acids, and water.

Le Chatelier has stated* the following to be the limits of these ingredients in good commercial cements:

Silica	21	to	24	per cent.
Alumina	6	"	8	"
Iron oxide	2	"	4	"
Lime	60	"	65	"
Magnesia	0.5	"	2	"
Sulphuric acid	0.5	"	1.5	"
Carbonic acid and water	1	"	3	"

Bleininger, in a chapter† on the Nature of Portland Cement, sets the following limits:

Silica	19	to	26	per cent.
Alumina	4	"	11	"
Iron oxide	2	"	5	"
Lime	58	"	67	"
Magnesia	0	"	5	"
Sulphuric acid	0	"	2.5	"
Alkalies	0	"	3	"

The percentages given by Le Chatelier should be considered as average rather than limiting values, since many good cements have an average composition largely out of these limits; Bleininger's limits, on the other hand, are somewhat extreme.

Table IV. gives the results of chemical analyses of many standard brands and serves to show the composition of commercial cements. In addition to these important ingredients, the following usually occur in very small quantities: Titanium, phosphoric acid, sulphur, manganese, silicious sand, coal and ash from the kilns, flint and iron from the mills, etc. The sum total of these elements, however, rarely equals one per cent.

*Transactions American Institute of Mining Engineers, 1893.

†"Manufacture of Portland Cements," by A. V. Bleininger. Fourth Series, Bulletin 3, Ohio State Geological Survey.

TABLE IV.—The Composition of Commercial Portland Cements Made from Various Raw Materials.

No.	LOCALITY	MATERIALS	Silica SiO_2	Alumina Al_2O_3	Iron Fe_2O_3	Lime CaO	Magnesia MgO	Sulphuric Acid SO_3	Alkalies $K_2O + Na_2O$	Loss on Ignition
* 1	Lehigh Co.—Penna....	Cement Rock and Limestone....	21.30	7.65	2.85	60.95	2.95	1.81	1.15	......
* 2	" " "	" " " "	19.66	7.47	2.29	61.23	2.83	1.34	1.41	......
* 3	" " "	" " " "	22.68	6.71	2.35	62.30	3.41	1.88	......	......
* 4	" " "	" " " " ...	24.48	4.51	2.68	64.33	2.59	1.41	......	......
† 5	Warren Co.—N. J....	" " " "	20.14	7.51	3.33	62.71	2.34	1.64	....	......
* 6	Greene Co.—N. Y........ ..	Limestone and Clay............	23.44	6.35	3.99	63.21	1.15	1.22	......	
* 7	Warren Co.—N. Y......	" " "	21.50	10.50		63.50	1.80	1.50	0.40	......
† 8	Craigsville—Va...........	" " "	21.20	7.90	......	63.14	2.40	1.37	......	...
† 9	Dallas—Texas.............	" " "	22.50	8.35	4.25	62.35	......	1.75	......	......
* 10	Onondaga Co.—N. Y......	Marl and Clay................	21.98	8.20	3.70	61.83	1.43	1.18	0.84	......
* 11	Syracuse—Ind.........	" " "	22.06	4.80	1.66	65.44	3.82	0.90	......	......
† 12	Coldwater Co.—Mich ...	" " "	21.22	'7.51	3.83	63.75	0.82	1.58	......	1.02
† 13	Sandusky—Ohio	" " "	23.08	6.16	2.90	62.38	1.21	1.66	......	...
† 14	Yankton—S. D..........	Chalk and Clay	19.50	7.70	4.30	60.00	0.80	2.80	1.20	......
* 15	Marengo Co.—Ala.........	" " "	19.56	12.16		62.27	0.64	0.54	......	..
† 16	Chicago—Ill.	Slag and Limestone	23.62	8.21	2.71	61.92	1.78	1.32	......	0.52
† 17	Youngstown—Ohio	" " "	22.84	12.77	......	51.30	2.64	0.48	.....	0.18
‡ 18	England.................	Chalk and Estuary Mud.......	22.10	11.32		60.76	1.10	1.40	......	1.94
§ 19	Germany	Chalk and Clay	24.90	8.00	3.22	59.38	0.38	1.46	0.50	2.16
§ 20	France..................	" " "	20.42	12.00	1.87	63.13	0.58	......	.. ..	2.00

AUTHORITY
- * ECKEL.—"Cement Materials and Industry."
- † BROWN—"Directory of American Cement Industry."
- ‡ TAYLOR & THOMPSON—"Concrete—Plain and Reinforced."
- § CUMMINGS—"American Cements."

Silica (SiO_2).—Silica constitutes from 19 to 24 per cent. of a Portland cement, and combined with lime to form calcium silicate furnishes the active factor in its hardening. It should exist entirely in a combined state and not as quartz sand, in which condition it does not combine with the lime, and is inert in the subsequent reactions.

Lime (CaO).—A well proportioned cement will contain from 59 to 67 per cent. of lime, depending upon the amounts of silica and alumina present. Provided all of it exists in a combined state, the greater its amount, the higher will be the strength of the product. If, however, more lime is present than will combine with the silica and alumina, an unsound cement will result, since the excess of lime in slaking will expand and so cause disintegration. The recent demand for high strength values at early periods has caused many manufacturers to produce very high limed cements, often passing the limit of safety unless exceptional care has been taken to so finely grind the raw materials and to burn so perfectly that the theoretical limit of combination can be approximated. A high proportion of lime also requires greater heat in burning, and forms a slow-setting cement.

*"Too low a proportion of lime, on the other hand, produces a fusible clinker, liable to overburning. This is especially the case with aluminous materials. If hard-burned, such mixtures give a fused clinker liable to fall to dust on cooling, hard to grind, and yielding slow-setting cement of poor hardening properties. If light burned, an over-clayed mixture yields soft brownish clinker, grinding to a brownish, quick-setting cement of inferior strength."

Alumina (Al_2O_3).—From 5 to 10 per cent. of alumina is usually present in Portland cement, mostly combined as calcium aluminates, and since the setting properties are due to these aluminates, the greater the proportion of this ingredient, the quicker will be the setting of the product. The ultimate tensile strength of high alumina cements is also apt to be inferior. The presence of alumina tends to facilitate the burning, since its compounds are much more fusible than those of silica, and if present in

*S. B. Newberry in "Concrete, Plain and Reinforced," by F. W. Taylor and S. E. Thompson.

excess make a mixture difficult to burn uniformly and hard to grind.

Iron Oxide (Fe_2O_3).—According to Le Chatelier, ferric oxide and calcium carbonate on burning yield products which slake with water and possess no hydraulic properties. Schott and Richardson, however, have prepared cements in which all of the alumina was replaced by iron, and therefore conclude that the function of these two ingredients is practically similar. The investigations of S. B. and W. B. Newberry also appear to show that the action of ferric oxide and alumina in promoting the combination of silica and lime is practically identical. The amount of iron usually present in Portland cements is less than 4 per cent., and it exerts but little influence on the physical properties of the material. The dark gray color of cement is due to the presence of iron compounds, cement prepared from silica, alumina and lime only being colorless.

Magnesia (MgO).—The role of magnesia in Portland cement has not yet been definitely determined. Certain investigators, including Le Chatelier, claim that magnesia may replace lime, and form silicates and aluminates of magnesia whose characteristics are similar to those of calcium. Others, including Erdmenger, Richardson and Newberry, consider that magnesia remains free in cement and is present only as an adulterant.

Investigation of two or three failures of important engineering works placed in sea water developed the fact that the concrete was high in magnesia, but it has been claimed that its presence was due to a deposition of magnesian salts from the water, and that the magnesia originally present in the cement was not responsible for the failures. A committee of the Association of German Portland Cement Manufacturers in 1895 presented a report stating that their investigations had shown that a content of magnesia up to 8 per cent. was harmless. Dyckerhoff, however, presented a minority report, stating that his experiments had shown that more than 4 per cent. of magnesia, whether added to a normal mixture or substituted for an equivalent portion of lime, caused a steady falling off in the strength of the cement, although actual cracking was observed only with 8 or more per cent. S. B. Newberry has stated that he has made a cement containing 9 per cent. magnesia which stood the boiling test, but that one containing 15 per cent. failed after several hours' boiling.

American Portland cements contain on an average from 2 to 4 per cent. magnesia, the latter value being the limit placed by the specifications* of the American Society for Testing Materials.

Sulphuric Acid (SO_3).—Portland cement contains usually from 1.25 to 1.75 per cent. of this ingredient, a large proportion of which is due to the admixture of calcium sulphate† with the finished cement for the purpose of regulating the setting properties. Such additions, however, should not exceed 2 or 3 per cent., which is equivalent to about 1 or 1.5 per cent. of the anhydrous sulphuric acid, since an abnormal amount is injurious to both the strength and soundness of the product, especially if placed in sea water. The standard French specifications limit sulphuric acid to 1 per cent., while American practice is to permit from 1.5 to 2 per cent. The specifications of the American Society for Testing Materials place the limit at 1.75 per cent.

Sulphur (S).—A small amount of sulphur is sometimes introduced from the coal in burning, and occasionally from the reduction of sulphates in the raw materials. These sulphides cause the cement in hardening to become mottled with dark blue spots, and if present in appreciable quantities may cause disintegration, due to the expansion in oxidizing on exposure to the air.

Alkalies (K_2O and Na_2O).—These appear to have but little influence on either the burning or quality of the cement. It has been held that alkalies acted more or less as a flux in facilitating the combination of lime with the silica and alumina, but later experiments have apparently disproved this theory. Excess of alkalies, under certain conditions, is said to be responsible for unsoundness, although this fact has never been definitely proven. The percentage of alkalies is usually from 0.5 to 2 per cent.

Carbonic Acid (CO_2).—Provided the temperature of burning has been sufficiently high, the presence of this element is due solely to absorption from the air, and since lime when insufficiently combined is very active in this absorption, an abnormal percentage of carbonic acid must show either underburning or an

*See Appendix C.
†See pages 26 and 83.

excess of lime. Normal cements contain from 0.5 to 1.5 per cent. of carbonic acid.

Constitution.—Although Portland cement has been extensively manufactured for well over half a century, it is only in comparatively recent years that a scientific study of the constitution of cement has been undertaken, and even at present the exact state of combination in which the different components exist has not been definitely ascertained. For many years it was the practice to proportion raw materials and to study the finished product by means of the "hydraulic modulus," or the ratio of the weight of lime to that of silica, alumina and iron, which varied between 1.8 and 2.2, but it soon became understood that the lime combined in different ratios with the other components, and the inadequacy of the formula was thus recognized.

Le Chatelier in 1887 was the first to attempt scientifically to explain the constitution of Portland cement.* Following petrographic methods, and examining with a microscope thin sections of clinker and hardened cement, he succeeded in separating them into two chief constituents, which later were named by Törnebohm alit and celit, two accessory constituents, belit and felit, and in addition an amorphous isotropic mass of matter. He considered alit to be composed of tri-calcium silicate, $3\ CaO\ SiO_2$, which is the active element in the hardening of cement, and which hydrates as follows:

$$3\ CaO\ SiO_2 + Aq = CaO\ SiO_2\ 2.5\ H_2O + 2\ Ca(OH)_2.$$

Cement also contains a tri-calcic aluminate, somewhat unstable, but setting rapidly in water, hydrating according to the formula:

$$3\ CaO\ Al_2O_3 + Aq = 3\ CaO\ Al_2O_3 12\ H_2O$$

Portland cement, according to Le Chatelier, therefore consists of tricalcium silicate mixed with calcium aluminate and ferrate and also containing mono and dicalcium silicates. He expresses the hydration of cement by the following two reactions:

$$2\ (3\ CaO\ SiO_2) + 9\ H_2O = 2\ CaO\ SiO_2\ 5\ H_2O + 4\ Ca(OH)_2,$$

and

$$3\ CaO\ Al_2O_3 + Ca(OH)_2 + 11\ H_2O = 4\ CaO\ Al_2O_3\ 12\ H_2O.$$

*Researches Experimentales Sur la Constitution Des Mortiers Hydrauliques—Annales des Mines—1887.

The calcium tri-silicate is produced by precipitation from a complex silico-aluminate, which permits the combination of the silica and lime.

He then fixes the amounts of lime and magnesia in Portland cements by the expressions:

$$\frac{CaO + MgO}{SiO_2 + Al_2O_3} \leqq 3$$

and

$$\frac{CaO + MgO}{SiO_2 - Al_2O_3 - Fe_2O_3} \geqq 3$$

in which chemical equivalents and not weights are used. Good commercial Portland cement nearly approaches the maximum of the first formula.

In 1897, S. B. and W. B. Newberry* prepared synthetically a number of compounds of silica, alumina and lime, which were thoroughly examined, and as a result of their investigations concluded that the essential constituents of Portland cement are tri-calcium silicate with varying proportions of di-calcic aluminate. This composition may be expressed by the formula:

$$X\ (3\ CaO\ SiO_2) + Y\ (2\ CaO\ Al_2O_3)$$

or substituting weights for equivalents, the formula becomes

$$\text{Lime} = \text{silica} \times 2.8 + \text{alumina} \times 1.1.$$

S. B. Newberry states†: "It is understood that this formula is merely empirical, representing the relative proportions present, since the aluminate remains for the most part in the magma in combination with part of the silica and with other substances."

"It should be remembered that this formula represents the *maximum* of lime which a Portland cement, burned in the usual manner, may contain without showing unsoundness. This maximum can be reached only by extremely fine grinding of the raw material. This formula, also, by no means represents the composition of finished cement, since the ash of the fuel lowers the lime and raises the silica and alumina, above that calculated from the raw material, by at least 2 per cent."

"In the laboratory, using gas as fuel, it will be found practicable to prepare sound cements corresponding to the above formula. In actual manufacture it is safer to reduce the lime

*The Constitution of Hydraulic Cements, by S. B. and W. B. Newberry, Journal of the Society for Chemical Industry, Vol 16, No. 11.
†In Taylor and Thompson's "Concrete, Plain and Reinforced."

slightly, to counterbalance possible defective grinding of raw material or unavoidable variations in composition. It will be found that the raw material at factories where the best Portland cements are made rarely falls below the composition,

$$\text{Lime} = \text{silica} \times 2.7 + \text{alumina} \times 1.0.$$

"This may be taken as a safe practical formula for commercial use."

In the past five or six years numerous investigators have worked on the problem of the nature of Portland cement, and many theories as to its constitution and hydration have been advanced, although it can be said that none of the essential theories based on Le Chatelier's original investigations have been positively disproved.

The subject of the constitution of cement should not be dismissed without allusion to Mr. Clifford Richardson's exhaustive researches. As a result of the examination of a series of synthetically prepared compounds, he arrives at the conclusion that the components of cement are present in a state of solid solution, and that alit and celit, which are the chief minerals found in Portland clinker, are composed—one of a solution of tri-calcic aluminate in tri-calcic silicate, and the other of di-calcic aluminate in di-calcic silicate. These, though miscible in the molten state, are not so in the solid form. The ratio of alit to celit may vary from 3 to 1 up to 6 to 1 and possibly over, depending on the relation of silicate to aluminate, and on the basicity of the clinker as a whole.

NOTE:—For further data on this subject, the reader is referred to Mr. Richardson's paper on "The Constitution of Portland Cement from a Physico-Chemical Standpoint," read before the Association of Portland Cement Manufacturers, June 15, 1904. A good resume of the different theories on the nature of cement will be found in a report on "The Manufacture of Portland Cement," by A. V. Bleininger—Fourth Series—Bulletin No. 3—Ohio State Geological Survey. Also a review of the literature on the subject prepared by Mr. Richardson was published in "Cement"—Volumes 4 and 5—Progress Publishing Company, New York.

CHAPTER III.

MANUFACTURE.

RAW MATERIALS.

Cement is composed essentially of a mixture of silica, lime, alumina and iron, so that any materials containing the proper proportions of these ingredients might be employed in the manufacture of Portland cement. In fact, Mr. Richardson has shown that, theoretically, a true Portland cement may be made by substituting other elements of the same groups for those ingredients which are considered to be essential, having made actual cements in which alumina is replaced by iron, lime by barium, and silica by tin, lead, and even phosphoric acid. From a commercial standpoint, however, only a limited variety of materials are adaptable.

The raw materials employed in the Portland cement industry in the United States may be divided into six classes:

(1) **Cement Rock and Limestone.**—Cement rock is an argillaceous limestone, low in magnesia, occurring chiefly in Lehigh Co., Pennsylvania, and Warren Co., New Jersey, although occasionally found elsewhere, as in the Virginias. Its composition averages from 35 to 40 per cent. of lime, and from 18 to 20 per cent. of silica. To produce Portland cement, an admixture of from 10 to 30 per cent. of pure limestone is generally required, although some manufacturers in the Lehigh District are so fortunate that they are able to make a proper mixture from different strata of rock occurring in the same quarry. About two-thirds of the Portland cement produced in this country is made from this combination of raw materials.

(2) **Limestone and Clay.**—These materials are most extensively employed in New York State, where clays averaging in composition about 55, 25 and 10 per cent. of silica, alumina and lime respectively are mixed with nearly pure limestone, the average mixture being about 20 to 30 parts of clay to 100 parts of limestone.

(3) **Marl and Clay.**—Marl is a soft, wet, calcareous earth,

almost a pure carbonate of lime, usually originating from shell deposits, although sometimes existing as the result of a chemical formation. It is found chiefly in the States of Ohio, Indiana and Michigan, but also occurs in New York and other localities. As with the second class, it generally requires an admixture of from 20 to 30 per cent. of clay. The ease with which the marl is excavated and reduced is the economical feature of this process, but this is counterbalanced by the handling of a large amount of water, and by the increased fuel consumption required for the burning of a wet mixture.

(4) **Chalk and Clay.**—This combination of raw materials is employed extensively abroad, but only to a limited extent in this country. Pure chalk, in fact, is never used in the United States, but a soft, chalk-like limestone, occurring principally in South Dakota, Arkansas and Texas, mixed with clay, is employed in a few mills.

(5) **Slag and Limestone.**—Certain blast furnace slags granulated and mixed with limestone may be burned to produce a true Portland cement. In Europe, slag has been utilized for this purpose for several years, but the process has only recently been introduced into this country. Sabin states* that, "whereas for the manufacture of slag (i. e., pozzuolana) cement, only the slag from gray pig iron is available, it is found that in most cases the slag from white pig iron may be used for the production of Portland cement from slag."

(6) **Alkali-Waste and Clay.**—These materials are used by a Michigan plant for the making of Portland cement, the waste occurring from the manufacture of soda by the ammonia-soda process, and existing as caustic lime, which is mixed with a suitable proportion of clay.

The average composition of these different groups of raw materials is shown in Table V., while Table VI. shows the extent to which each group is employed in the industry of this country.

The prime requisites for the suitability of any combination of materials are that the content of lime, silica and alumina is such that the resulting cement will contain these ingredients within the limits given in Chapter II.,† that the percentage of mag-

*In "Cement and Concrete," by L. C. Sabin, p. 22.
†See page 7.

TABLE V.—Showing the Composition of the Different Groups of Raw Materials, with the Cements Made from Them.

	1 Cement Rock and Limestone Lehigh Co.—Penna.			2 Limestone and Clay Eastern New York			3 Marl and Clay Central New York			4 Chalk and Clay South Dakota			5 Slag and Limestone Chicago—Ill.			6 Alkali Waste and Clay Michigan		
	Cement Rock	Limestone	Cement	Clay	Limestone	Cement	Marl	Clay	Cement	Chalk	Clay	Cement	Slag	Limestone	Cement	Alkali Waste	Clay	Cement
Silica.............. SiO_2	19.06	1.98	21.30	55.27	3.30	21.50	0.26	42.85	21.98	4.14	61.53	19.5	35.60	2 00	21.40	0.98	46 81	23.20
Alumina......... Al_2O_3	4.44	0.42	7.65	28.15	1.30	10.50	0.21	13.51	8.20	1.81	20 74	7.70	12.56	1.50	7.15	1.62	14.21	8.00
Iron .... Fe_2O_3	1.14	0.28	2.85				0.01	4.49	3.70	2.72	4.01	4.30			2.25			2.40
Lime..............CaO	38 78	53.31	60.95	5.84	52.15	63.50	50.98	12.69	61.83	51.00	5.28	60.00	48.09	95.00	62.42	50 40	14.04	62.10
Magnesia ....MgO	2.01	0.97	2.95	2.25	1.58	1.80	0.19	3.30	1.43	0.10	1.72	0.80	1.75	1.50	2.09	4.97	3 61	2.00
Sulphuric Acid......SO_3	...	....	1.81	0.12	0.30	1.50	....	....	1.18	0 50	1.26	2.80	....	....	1.50	....	1.18	....
Carbonic Acid......CO_2	32 66	42.94	....	....	40.98	....	40.26	13.59	....	37.99	3.09	....	....	....	....	....	....	...
Alkalies....$K_2O + Na_2O$	....	....	1.15	....	....	0.40	....	....	0.84	....	....	1.20	....	....	....	0.50	3 04	....
Organic and Water.....	....	....	....	...	8.37	....	7.98	....	....	....	....	....	...	....	....	....	....	....
Sulphur .... S		....	....	....	....	....	....	....	....	....	....	....	....	....	....	0 06	....	...

AUTHORITY.—(1)—Cement Materials and Industry of U. S.—E. C. Eckel—Bull. U. S. Geol. Survey, No. 243.
(2)— " " " " " " " " " " " " "
(3)— " " " " " " " " " " " " "
(4)— " " " " " " and Directory of American Cement Industries.
(5)— Personal Correspondence—Mr. E. M. Hagar, Illinois Steel Co.
(6)—B. B. Lathbury—in ENGINEERING NEWS—June 7, 1900, and Sabin's "Cement and Concrete."

nesia and ferric oxide in the finished product will be less than 3 and 4 per cent. respectively, that the content of sulphur be as low as possible, not over 1 per cent. in any form, the presence of

TABLE VI.—Showing Production of Portland Cement from Various Materials. (From article by E. C. Eckel, in ENG. NEWS, April 16, 1903.)

	Cement Rock and Limestone		† Limestone and Clay	
	Barrels	Total Per Cent.	Barrels	Total Per Cent.
1898 production	2,682,304	74.9	315,608	8.8
1899 "	4,010,132	70.9	458,000	8.1
1900 "	5,919,629	70.3	874,715	10.4
1901 "	8,503,500	66.8	1,710,773	13.5
1902 "	10,600,000	67.9	2,100,000	13.5
	*Marl and Clay		Chalk and Clay	
	Barrels	Total Per Cent.	Barrels	Total Per Cent.
1898 production	545,372	15.2	39,000	1.1
1899 "	1,095,934	19.4	88,200	1.6
1900 "	1,444,797	17.1	184,400	2.2
1901 "	2,001,200	15.8	495,752	3.9
1902 "	2,200,000	14.1	700,000	4.5

* Including also the product from alkali waste and clay.
† Including also the product from slag and limestone.

sulphides being particularly injurious, and that they are free from sand or silica in such a form that it will not enter into proper combination.

Calculation of Mixtures.—Since the composition of Portland cement can only vary between very close limits, it is evident that the chemical laboratory plays a most important role in the process of manufacture. Analyses of the raw materials, of the mixture before burning, and of the finished product are made at least once a day, and generally more often.

In starting a mill, or experimenting on new materials, formulas such as Newberry's* are frequently employed to proportion the mix. As an example, suppose it was desired to obtain the proper mixture of cement rock and limestone, analyzing as follows:

	Cement Rock.	Limestone.
Silica (SiO_2)	19.1	2.0
Alumina (Al_2O_3)	4.5	0.6
Lime (CaO)	38.8	53.3
Other ingredients	37.6	44.1
	100.0	100.0

the formula being,

$$\text{Lime} = 2.7 \text{ silica} + 1.0 \text{ alumina}.$$

*See page 14.

The silica and alumina in the limestone require $2.7 \times 2.0 + 1.0 \times 0.6 = 6.0\%$ lime, which, subtracted from 53.3, leaves 47.3% available for combination.

The silica and alumina in the cement rock require $2.7 \times 19.1 + 1.0 \times 4.5 = 56.1\%$ lime, but as only 38.8% is present, 17.3 parts of silica and alumina are unprovided for.

100 parts of cement rock will therefore require $17.3 \times 100 \div 47.3 = 36.6$ parts of limestone.

Several other formulas,* including the hydraulic index,† are also employed for this purpose. In the actual control of a mill, however, it is customary, provided the run of the materials is fairly uniform, to proportion by the percentage of lime alone. Suppose it has been found by experience that the best results are produced from a mixture containing 75% carbonate of lime, and that it is desired to obtain the proper mixture of a clay, and a limestone, containing 24% and 91% carbonate of lime respectively. It is then obvious that $91 - 75 = 16$ parts of clay, with $75 - 24 = 51$ parts of limestone, or 31.4 parts of clay to 100 of limestone will give the proper mixture. The same method of course applies equally well when the oxides instead of the carbonates are known.

Occasionally mixtures are proportioned from the percentages of silica, because of the greater ease with which this ingredient is determined chemically, but the method is much less accurate and is apt to give unsatisfactory results.

PROCESSES.

The various processes by which Portland cement is manufactured are commonly classified as dry or wet according to the method by which the raw materials are mixed and reduced, while a second classification divides them into rotary or stationary kiln processes according to the manner of burning. Each one of these methods consists essentially of three steps —the mixing and grinding of the raw materials, the calcining of the mixture, and the final pulverization of the clinker, the last of which is practically the same no matter what process is used, although the actual appliances may be very different.

*For discussion of several formulas on cement batch calculation the reader is referred to "The Manufacture of Portland Cement," by A. V. Bleininger—Fourth Series—Bulletin 3—Ohio State Geological Survey.

†See page 12.

The details of manufacture, however, vary considerably in different localities and are dependent both on the nature of the raw materials and on other local conditions, so that although the general methods are subject to classification, the details are rarely alike in any two mills.

Dry Process With Rotary Kilns.—This process is adaptable to any class of raw materials which can be mined and reduced

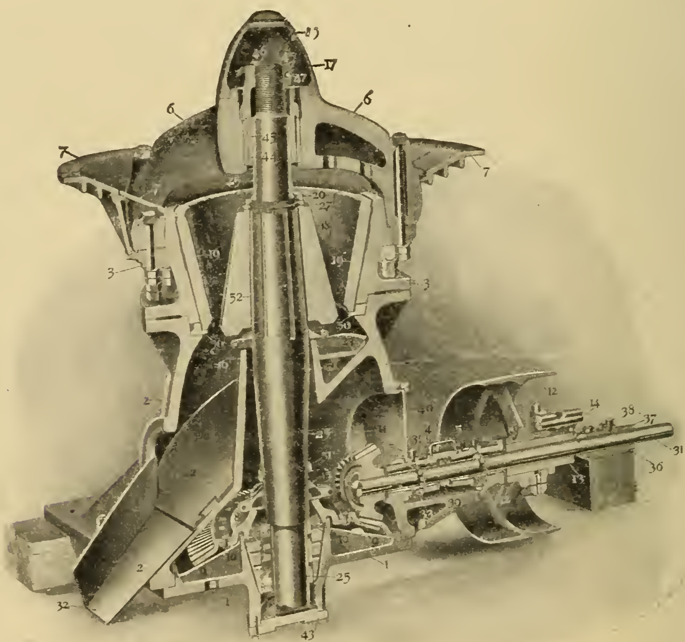


FIG. 2 —Spindle Crusher of the Gates Type.

in a dry condition. The cement-rock and limestone mixtures employed in the "Lehigh" district, as well as most of the limestone and clay cements are, in this country, treated almost exclusively by this process, over 80 per cent. of the American production of Portland cement being manufactured in this manner. In brief, the process is as follows:

The raw materials, after being mined or quarried, and conveyed to the mill, are first passed through crushers, either of the jaw or spindle type (Fig. 2), and reduced to a maximum

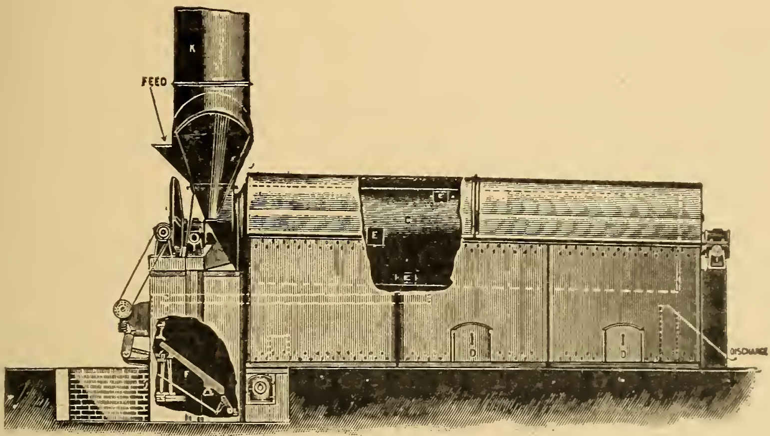


FIG. 3.—Rotary Drier of the Cummer Type.

diameter of 2 or 3 inches, after which they are sent to storage bins, where they are kept until their chemical composition has been determined so that the mix may be properly proportioned. The suitable mixture is then made on scales, which commonly are of the automatic type, and conveyed to a drier which is maintained at a temperature sufficient to drive off the

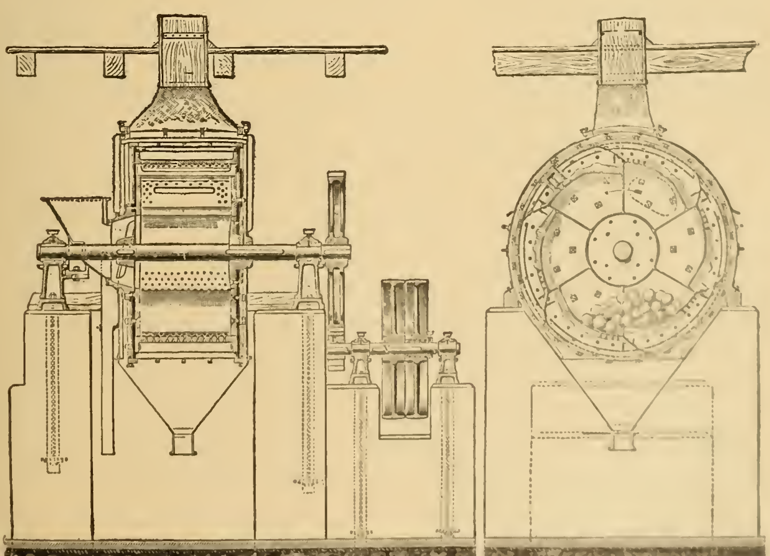


FIG. 4 —Ball Mill of the Krupp Type.

greater part of the moisture contained in it. These driers are usually built in the form of a rotating cylinder (Fig. 3) 4 or 5 feet in diameter, 40 or 50 feet long, and slightly inclined to the horizontal, the materials entering at the upper or stack end, and being discharged hot at the lower end. A small furnace usually furnishes the heat, although it has often been attempted to utilize the waste heat from the kilns.

From the drying cylinder, the materials are conveyed to a preliminary grinding machine of which the ball mill is the most common type, where they are reduced to a size small enough to pass a 20 or 30-mesh screen. The ball mill (Fig. 4) consists essentially of a revolving drum 5 to 8 feet in diameter, contain-

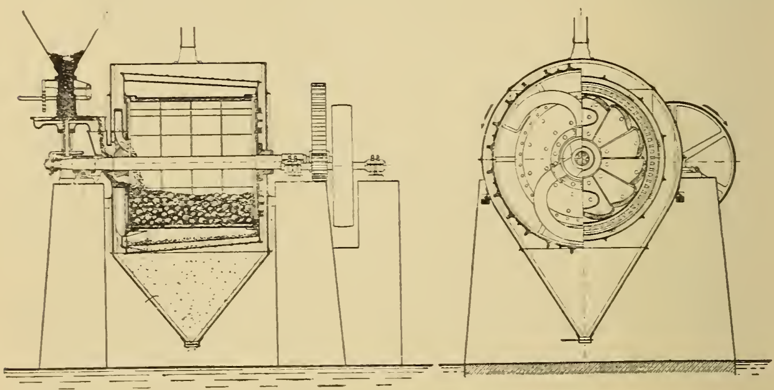


Fig. 5.—The Kominuter.

ing a charge of 3 to 5-inch steel balls, the periphery of the drum being made of perforated plates, overlapping each other, through which the ground material falls on screens, that of sufficient fineness passing through to a hopper, while the tailings return to the grinding chamber through the spaces between the plates. Other forms of preliminary grinding machines are the "Kominuter" (Fig. 5), an improved form of ball mill; rolls (Fig. 6) in which the crushing is performed between two rolls revolving towards each other and pressed together by heavy springs and the "Kent" mill (Fig. 7) which consists of a revolving ring with three rolls pressing against its inner face, the material being held to the tire by centrifugal force. Dis-

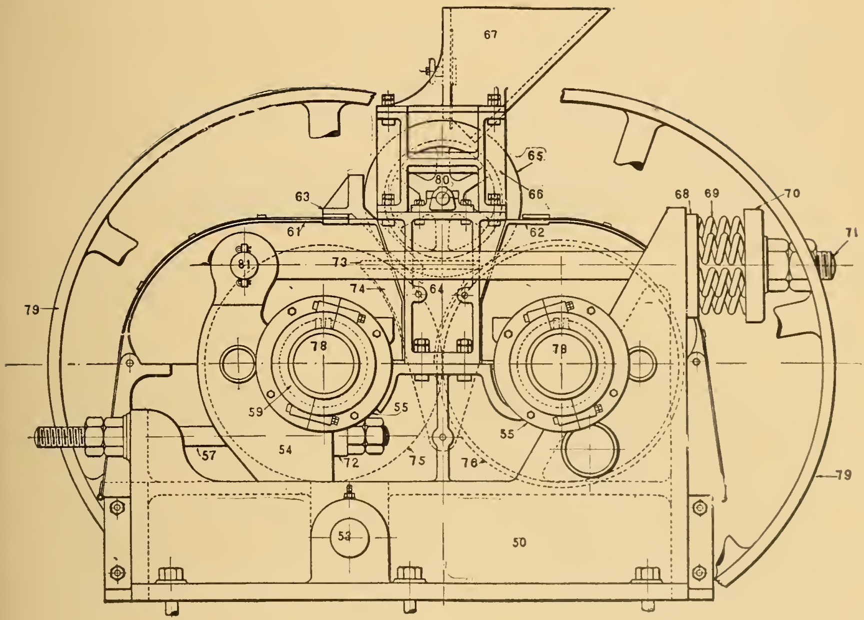


FIG. 6.—Buchanan Crushing Rolls.

integrators of the hinged hammer type are also occasionally employed.

After the preliminary grinding, the mixture of raw materials is passed to a fine grinder, where it is finally reduced to a size such that from 90 to 95 per cent will pass a No. 100 screen, or to about the fineness of the finished product. The most common types of fine grinding machines are the tube mill (Fig. 8) and the Griffin mill (Fig. 9). The tube mill consists essentially of an horizontally rotating cylinder 16 to 24 feet long and 4 or 5 feet in diameter, about half filled with flint pebbles, the

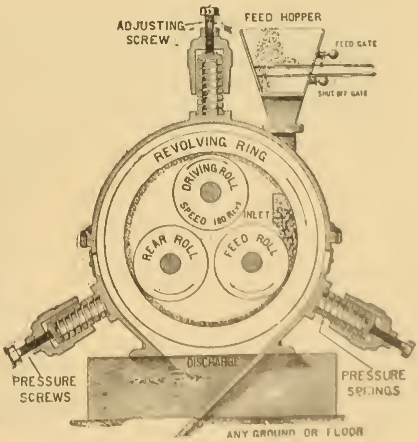


FIG. 7 —The Kent Mill.

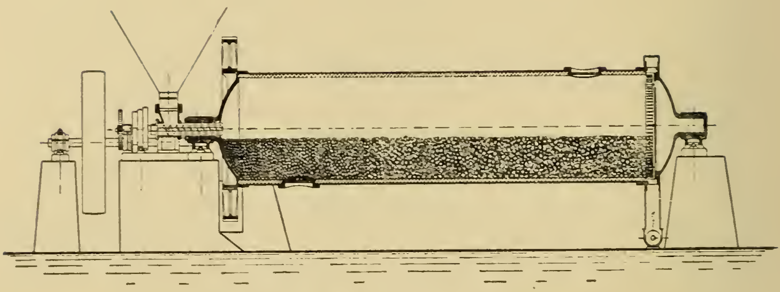


FIG. 8.—Tube Mill of the Davidsen Type.

material entering at the center of one end and leaving at the other. Ball and tube mills are commonly arranged in batteries, one ball mill supplying sufficient material for two of the tube mills. The Griffin mill is an ingenious device, the grinding being performed between a fixed circular tire, and a vertically rotating grinding disk suspended from a universal joint, which is pressed by centrifugal force against the tire around which it turns in a direction opposite to that of its rotation, the fine material passing out through vertical screens, while the coarser particles are caught up by the shoes under the disk and again acted upon. Although these two mills are the most common, several other forms may be employed. Mills like the "Kent" are sometimes, in connection with an air separator, made to perform the final grinding. Rolls also may be used for this reduction, the Edison plant employing this form of grinder exclusively for both crushing and pulverizing.

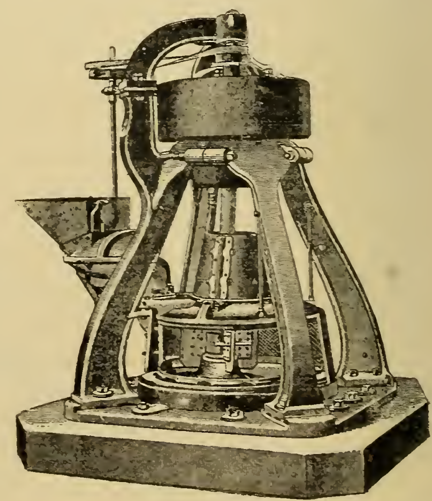


FIG. 9.—Griffin Mill.

This method of handling the raw materials is frequently varied, chiefly in two particulars: First, when raw materials of similar texture, such as cement-rock and limestone, are employed, the mixture is often made prior to the preliminary crushing so that the materials are never handled separately, and, second, when very dissimilar materials, like limestone and clay, are used they are usually crushed, dried, and coarsely ground separately and the mixture made only before passing to the final grinding. Clay and marl may be handled by this last

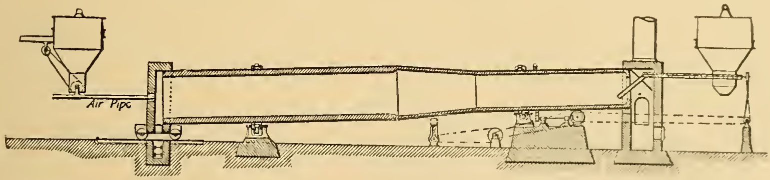


FIG. 10.—Cross-Section of a Rotary Kiln.

method, although the wet process has usually been found more economical for this combination. The distinctive features of the process, however, are similar for all materials.

From the grinding machines the mixture is conveyed to bins above the rotary kilns into which it is fed automatically. The rotary kiln (Figs. 10 and 11) is a steel cylinder varying in length from 40 to 150 feet, and from $4\frac{1}{2}$ to 9 feet in diameter, lined with from 6 to 12 inches of fire-brick, inclined 8 or 10

FIG. 11.—Rotary Kiln as Made by The Bonnot Co.

degrees to the horizontal, and arranged to rotate at a speed averaging about one turn per minute. The raw materials enter at the upper end in the form of a powder and in passing through are calcined to a clinker which leaves the kiln in small balls ranging from $1\frac{1}{2}$ inches to $\frac{1}{8}$-inch in diameter. The fuel generally employed is finely powdered gas slack coal, although both oil and producer gas have also been used. The coal is blown into the lower end of the kiln by a fan or compressed air and instantly ignited, forming a flame which reaches from 15 to 25 feet into the kiln, and which creates a temperature of from 2,600° to 3,000° Fahr. The pulverization of the coal is performed in tube or "Griffin" mills or in disintegrators, after having first been dried, and is so finely ground that about 90 per cent. will pass a No. 100 sieve. The temperature of burning, and the time required for it vary considerably with the character of the materials and of the fuel as well as with other conditions, but the temperature will average about 2,700° or 2,800° Fahr., while about half an hour is the average time required for the materials to pass through an ordinary kiln of about 60 feet in length. Well burned clinker is of a greenish black color, of a honey-combed structure showing traces of fusion, and is fairly uniform in size.

On leaving the kiln, the clinker is sprayed with a small stream of water which both cools it, and makes it more easy to pulverize, and then is passed through coolers which reduce the heat to normal. These coolers are either constructed in the form of a revolving cylinder, similar to the driers for raw materials, the clinker passing through against an air blast, or in the form of a vertical stationary cylinder through which the clinker works its way down over a system of baffle plates against a strong current of air. After cooling, the clinker should be stored for some little time before the final grinding, since the seasoning of the expansives is apparently most active at this time.

The final reduction is effected by passing the clinker through batteries of coarse and fine grinding machines such as ball and tube mills, rolls and Griffin mills, Kent and tube mills, or some similar combination of these pulverizers. Recourse is frequently made to air separation of the powder, as an economical feature of the process, and, with these separators, mills such as the Kent may be used alone for the final reduction, thus dispensing with the second machine. The finished cement, finally, is conveyed to the stock house and, after a further storage, is packed in bags or barrels for shipment.

The addition of sulphate of lime, either in the form of gypsum or plaster of Paris, is usually made while the clinker is passing from the coarse to the fine grinding machine, although, in some mills, the admixture is made in the stock house, imme-

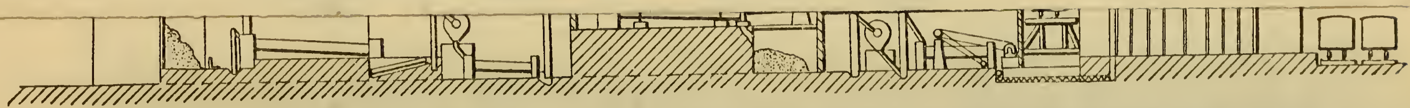


FIG. 12. PLAN AND SECTION OF A PORTLAND CEMENT MILL USING THE DRY PROCESS WITH ROTARY KILNS.

(To face page 27.)

diately before packing. The first of these methods secures a more thorough mixture, although the sulphate is apparently more effective when the latter method is followed, the long storage at the high temperature of the bins seeming to affect its activity.

The plan of the cement mill shown in Fig. 12, illustrates the general lay out of the various machines and appliances used in this method of manufacture. The dry process with rotary kilns may be considered the typical American method for the manufacture of Portland cement.

Dry Process With Stationary Kilns.—This method, at one time, was largely employed in the United States, but has now been almost superseded by the rotary process. The advantage of the process is the reduced fuel consumption, which, however, is usually more than off-set by the increased amount of labor required. The relative economy of the two processes thus depends upon the relative cost of fuel and labor, in this country the rotary process being generally found the more economical, while, in certain foreign countries, the high cost of fuel combined with the cheapness of labor, often creates the reverse condition. The quality and properties of the cement made by the two processes are very similar, although that produced in rotary kilns is apt to be more uniform.

In this method, the raw materials are mixed, dried and ground in a manner similar to that already described, but after the final grinding the material is passed through a pug mill where it is mixed with a small amount of water, then pressed and cut into bricks or cubes, the process being very similar to that employed in the ordinary making of building brick, after which they are sent to drying tunnels where they are dried and hardened.

From the tunnels, the bricks are taken to the kilns where they are burned to a clinker. The more uneven character of the burning in stationary kilns usually makes imperative a sorting of the clinker, and a discarding of that which is either under or over burned, after which the process is similar to that employed with rotary kilns, except that the larger size of the clinker usually requires a preliminary rough crushing before it is sent to the grinding machines. A plan of a cement mill using this process is shown in Fig. 13.

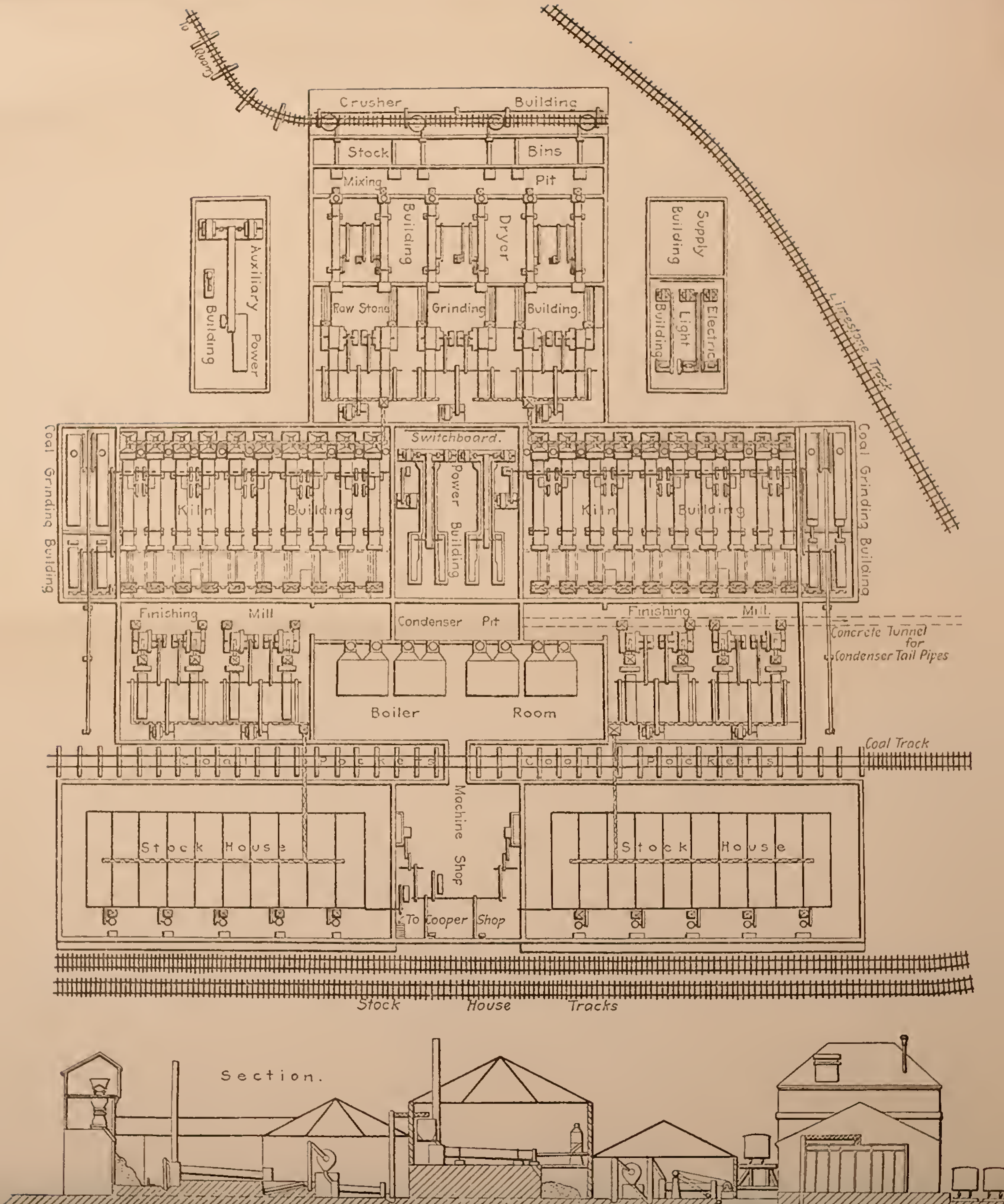


FIG. 12. PLAN AND SECTION OF A PORTLAND CEMENT MILL USING THE DRY PROCESS WITH ROTARY KILNS.

(To face page 27.)

Stationary kilns are of three general types—the dome kiln, the continuous or shaft kiln, and the ring or chamber kiln. The dome kiln consists of a single shaft in the form of an inverted bottle in which the bricks of cement materials and the fuel, usually in the form of coke, are placed in alternate layers, and then fired and burned. After cooling, the clinker is drawn from the bottom, sorted for the purpose of discarding that poorly burned, and then ground to cement. The use of this kiln is uneconomical in the heat wasted when firing and when cooling, and also in the comparatively small output, due to the

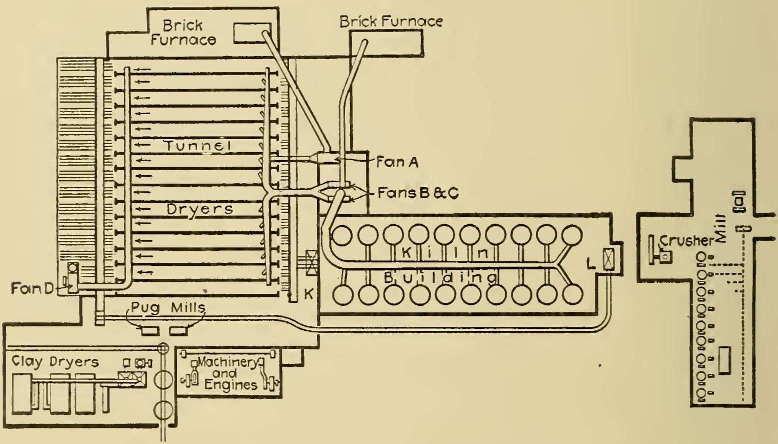


FIG. 13.—Plan of the Works of a Portland Cement Plant Employing Stationary Kilns.

intermittent character of the operation. The burning also is much more difficult to control so that the poorly calcined clinker amounts to a considerable proportion of the output. Its use is commonly limited to mills of very small capacity.

Continuous or shaft kilns are of several types, among which the Aälborg, Dietzsch and Schoefer are the best known, but are all similar in that they consist of a tall vertical shaft into the upper part of which the materials and fuel are charged in alternate layers, while the finished clinker is drawn from the bottom. Most of these kilns are contracted near the center at the point where the temperature reaches its maximum. The kiln illustrated in Fig. 14 is an American adaptation of the Schoefer type used in one of the few mills that still follow this process.

Ring or chamber kilns consist of a series of chambers arranged around a central stack in such a manner that while the materials are being burned in one chamber the exhaust heat passes through the other chambers, thus raising the temperature of the materials about to be burned. This system is well

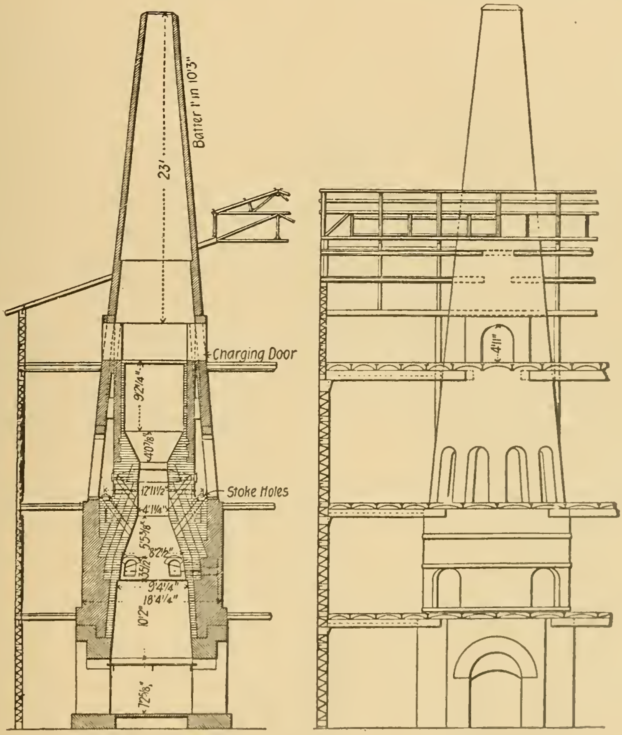


FIG. 14.—Continuous Kiln of the Schoefer Type.

known in the brick industry, but is not employed in this country for the burning of cement.

Discussing the relative economy of dome and continuous kilns as compared with those of the rotary type, Mr. F. H. Lewis, in "The Cement Industry," gives the following data:

Quantity of fuel required per day:

Intermittent kilns	15 to 30 barrels.
Continuous shaft kilns	40 to 80 barrels.
Rotary kilns	120 to 250 barrels.

Ratio of fuel consumed to clinker produced:

Intermittent kilns	25 to 35 per cent. (coke).
Continuous shaft kilns	12 to 16 per cent. (coal).
Rotary kilns	30 to 40 per cent. (coal).

Comparison of cost under American conditions:

	Rotary kiln.	Continuous shaft kiln.
Labor cost per barrel	$2\frac{1}{4}$ to 4 cents.	12 to 14 cents.
Fuel cost per barrel	11 to 15 cents.	5 to 6 cents.

Wet Process With Rotary Kilns.—In the United States, this process is utilized only by the mills (see Fig. 15) operating on marl, with the exception of the one mill which utilizes the waste from the manufacture of soda, but it also may be applied to any other raw materials, such as chalk, that exist in a finely divided state, although not excavated in a wet condition. Assuming the use of marl and clay, the process is as follows:—The marl, after excavation, is passed through a disintegrator and sometimes a stone and grass separator, and run into storage basins, while the clay is dried, pulverized, and then mixed with a proper amount of marl in pans of the edge runner type (Fig. 16), the slurry containing enough water to give it a thick creamy consistency. In some mills, this process is varied by mixing the clay with water before adding it to the marl. The mixture then is ground, while still in a wet condition, in either edge runners or tube mills, from which it is run into slurry tanks where it is kept agitated by revolving paddles or by compressed air, and where chemical analyses are made to check the accuracy of the proportions, corrections being made if necessary. Centrifugal pumps and compressed air are both used for handling the slurry.

The wet slurry is then pumped directly into the upper ends of rotary kilns which usually are somewhat longer than those employed in the dry process so that the waste heat may be utilized in driving off the excess water. About 150 to 160 pounds of coal per barrel of cement are necessary for the burning, which is 30 to 50 per cent. more than is required in the dry process, but this disadvantage is largely compensated by the cheaper method of handling and preparing the raw materials. The treatment of the clinker is similar to that of the other processes.

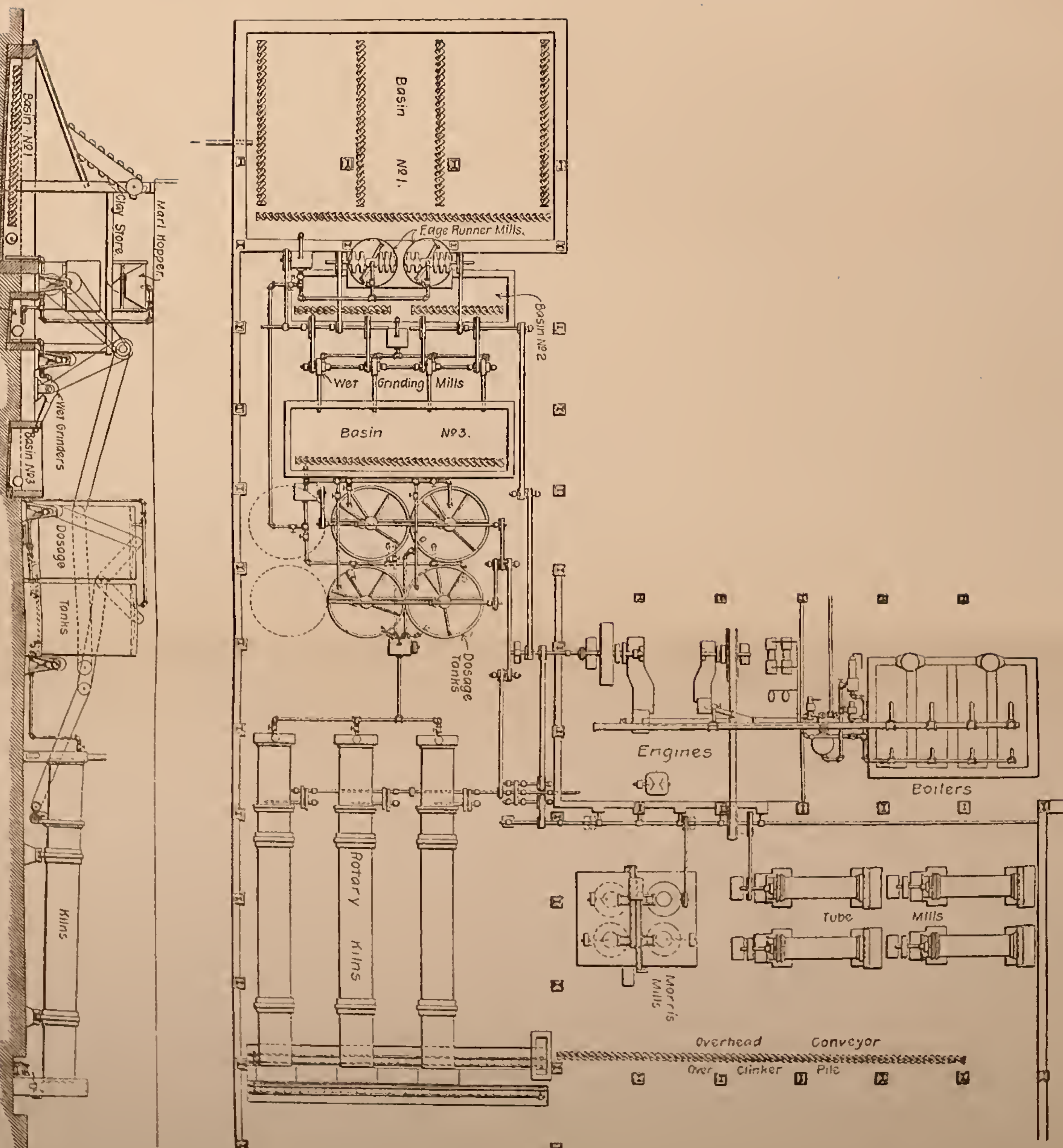


FIG. 15. PLAN OF THE WORKS OF A PORTLAND CEMENT MILL USING THE WET PROCESS.

(To face page 30.)

Wet Process With Stationary Kilns.—This process also is only adaptable to soft, wet, or finely divided materials. The clay and marl, or chalk, are first ground, if necessary, and then mixed together in a wash mill with a large excess of water, the lumps being broken up by means of agitators. When the materials have thus been reduced to a very finely divided state,

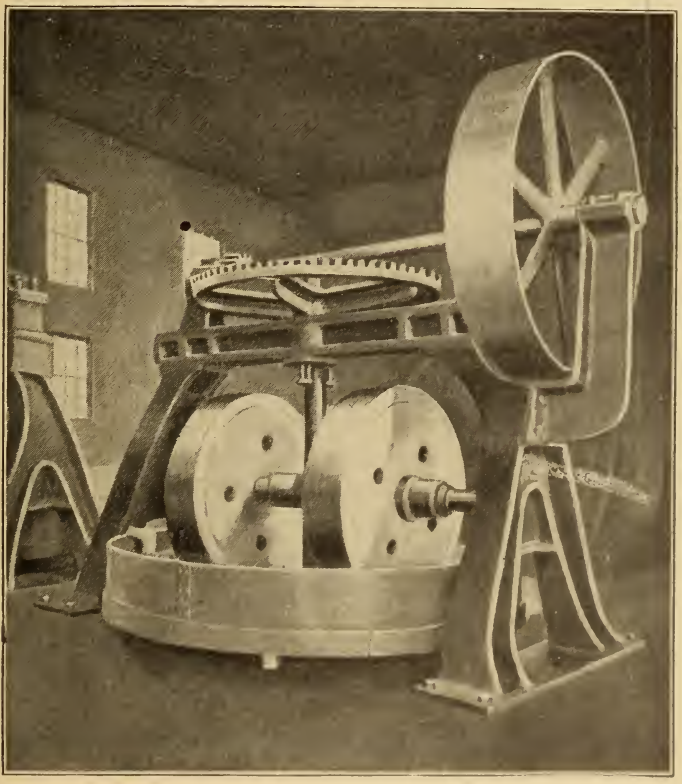


FIG. 16.—Mixing Pan for Marl and Clay.

the mixture is run into settling basins, where the solid matter settles and from which the excess of water is drawn off. The slurry when still further hardened is then formed into bricks, and burned in stationary kilns.

A modification of this method, known as the semi-wet process, consists in mixing with a smaller amount of water, sufficient to give a creamy consistency, the operation being similar to the wet process with rotary kilns except that the slurry is partly dried and formed into bricks instead of being fed directly into the kilns.

The chief disadvantages of the process are the large space necessary for settling and drying the slurry, and the greater amount of labor required. It, however, is used extensively in Europe, and in England a few years ago might have been considered the typical process. In this country, cement is not made by this method.

Portland Cement From Slag.—The only other distinctive process employed in the United States is in connection with the utilization of blast-furnace slag. The slag immediately after leaving the furnace, is sprayed with a stream of water which not only granulates it, but also drives off the sulphur, changing the calcium sulphide into calcium oxide and hydrogen sulphide which is evolved as gas. The slag is then dried, ground, mixed with the proper amount of ground limestone and burned in rotary kilns, following the dry process.

Essentials to Good Manufacture.—From the standpoint of the production of well-made material, in contradistinction from that of economy, the essentials to good manufacture are:

(1) Proper raw materials; so that a mixture containing the correct proportions of silica, lime, alumina and iron may be made from them, and also containing but a small percentage of the injurious constituents notably magnesia, sulphur, and the alkalies.

(2) Correct proportioning; it being impossible to produce good cement from an incorrectly proportioned mixture.

(3) Fine grinding of the raw materials. When the raw materials are calcined, the formation of the different constituents of cement takes place by a process of diffusion, so that only those particles which exist in a very finely divided state are capable of combining properly. The most common cause of unsoundness in cements is insufficient grinding of the raw materials.

(4) Proper burning. If the temperature of burning is too high or too low, or if the duration of the calcination is either lengthened or shortened, the character of the product

will be vitally affected, and if varying beyond a very limited range the material suffers exceedingly in quality.

(5) Sufficient storage. Both the clinker before final grinding and the finished cement should be kept in storage for a considerable time in order that any expansives that may be present will have sufficient time to absorb water and carbonic acid and thus become inert. Most cements require from 2 weeks to a month for this action to take place, and should never be used in less than that time. As a rule, the more finely the raw materials are ground, the less time is necessary for storage.

CHAPTER IV.

INSPECTION AND SAMPLING.

Reception and Storage.—Portland cement is shipped from the manufacturers to the site of the construction work either in barrels of wood or in bags of cloth, canvas or paper, the net contents of four of these bags equaling that of a barrel. Shipments intended for distant points, especially when carried by water, are usually sent in wood, but under ordinary conditions bags are generally employed. In other words, barrels are employed when the material may be subjected to excessive dampness, or when it will not be used for a considerable time, since the cement is better protected when thus packed, but, on account of the much greater ease in handling as well as being more economical, bags are used whenever it is practicable. From 85 to 90 per cent. of the domestic product is shipped in bags.

The common cloth bag has the advantage of being adaptable to much rougher handling without danger of destroying it, but on the other hand paper bags are cheaper and have the additional advantage of being broken and so rendered unfit for filling a second time after the material has been removed, and thus make rebagging on the part of local dealers impossible. Owing to the fact, however, that the paper bags are often broken in transportation and in handling on the work they are justly rather unpopular and hence used but infrequently. The danger of allowing an unprincipled dealer to rebag an inferior quality of cement in packages marked with a standard brand may be obviated by requiring the manufacturers to seal each of their bags with a wire and stamped lead seal, which is destroyed when the package is opened. The additional expense of this sealing is insignificant, and should be required on any important piece of construction where the material is not purchased from the manufacturers directly, and when sufficient facilities for testing are not at hand.

It is customary to inspect and test cement after it has been received on the site of construction, both as a matter of convenience, and to ensure against the substitution of inferior material after inspection, although the practice on two or three of the most important engineering works, notably that of the New York Rapid Transit Subway, has been to inspect the material in the stock house at the manufacturers. For works using an extremely large quantity of material produced at a single mill this may be advantageous, but otherwise it is impracticable, and furthermore is less accurate for the reason that the conditions surrounding the cement during transportation may be of such a nature as to alter its physical properties completely, so that the material tested is radically different from that used.

The specifications for the reception of cement shipments usually stipulate a definite time, never less than eight or ten days, during which they must be held on the work while undergoing test, and this necessitates ample facilities for storage. The principles for storing cement properly are first to protect it from dampness or excessive heat, and secondly to allow the access of as much dry air as possible. Cement is generally received in car-load shipments of from 100 to 150 barrels, and it is convenient to inspect, test and store the material in these car-load lots. Store houses, therefore, should be divided or partitioned into a number of bins, each being of size sufficient to hold one car load and so arranged that each bin is readily accessible so that it may be filled or emptied with a minimum amount of labor. Over, or by the side of each bin, should be placed a board or placard on which is written the brand of cement, the number of packages, the name and number of the car in which it was shipped, and the date when received, and after test should be marked accepted or rejected, with the date. Rejected material should be removed at once under the supervision of an inspector, and some or all of the packages marked with a private mark so that it can be recognized if attempt is made to ship it back again.

When using standard brands of cement with which the engineer has had considerable experience, a seven-day test is usually sufficient. New or unfamiliar brands should, however, never be accepted on less than a twenty-eight-day test. Cement failing in certain tests may, by reason of the additional seasoning gained in two or three weeks, pass those tests at the expiration of that time, so that the retesting of cement failing in the first tests is perfectly justifiable in certain instances, and, moreover, the fact that there may be a wide discrepancy between the results of the two series of tests need not necessarily cast discredit on the laboratory.

Inspection.—The field inspection of cement shipments should include an examination of the condition of the packages and of the material, examination of the storage facilities, and, if required, a determination of the average weight of the packages.

The packages should each be plainly marked with the brand and name of the manufacturer; unbranded packages should be discarded and not allowed to enter the work. They should be in fairly good condition, securely tied, and, if so stipulated in the specifications, sealed properly with a lead seal. In case the seal and brand mark bear different names, the name on the seal should govern, but this should not be allowed to occur except in occasional instances.

Regarding the condition of the cement as a whole, the field inspector often is able to form a more correct judgment than the tester in the laboratory. Old or well seasoned cement generally appears rather lumpy, but these lumps can easily be crushed in the fingers and hence in making the mortar or concrete are entirely broken up and thus are not detrimental. If the material, however, has been subjected to excessive dampness, or has actually been wet, lumps are formed, similar in appearance to those occurring in old cement, but are hard and can only be crushed by the exertion of considerable force. Although when a concrete machine mixer is used these lumps may be well broken up, the cement in them is, nevertheless, partially hydrated and hence inferior. In hand mixing the making of a smooth mortar from such cement is almost impossible. Material containing lumps of this character is occasionally screened and the siftings used, but even then the finer particles, which must have been subjected to nearly the same conditions as those that formed lumps, cannot be of as good a quality as originally. It is, therefore, usually advisable to reject outright a shipment containing any appreciable quantity of these hardened lumps, unless, of course, the conditions producing this result only affect a certain part of the shipment, in which case only that part need be rejected.

The color of Portland cement* affords no criterion of quality in field inspection, except in so far as uniformity is concerned. If it is observed that the contents of different packages are different in color, it is obvious that the shipment is not all of the same material and tests should be made from separate packages to ascertain whether the cement is all acceptable, or whether it is a mixture of good and bad material.

The store-house should be inspected to see whether the material is properly protected, so that it is impossible for the cement to deteriorate in quality while it is being held. It frequently happens that a shipment may show excellent tests but, by the time of their completion, the material has been so mishandled as to have become worthless.

A provision of many cement specifications† is that the net weight of the packages shall not be less than a certain fixed quantity. This determination being made in the field is considered as part of the inspection rather than the testing and consists of weighing, say 10 packages, either separately or together, and then the bags or barrels after the material has been emptied from them, the difference being the net weight of the packages.

A full report of the field inspection of every shipment should be sent to the testing laboratory with the sample and made a part of the permanent records.

Sampling.—The maximum size of a shipment of cement which can be represented by a single sample for testing, is a matter governed more by local conditions and the discretion of the engineer than by any fixed rules. In practice, since cement is usually shipped in car-load lots of 100 to 150 barrels, it is convenient to represent this quantity by a single sample, but this quantity is near to the safe maximum, so if a lot of cement exceeds 150 barrels, it is advisable to separate it into portions of not over this amount, and to sample each portion separately.

The sample for testing is generally taken in one of three ways: (1) An average sample from several packages; (2)

*See page 10.
†See Appendices.

separate samples, each from a single package, tested separately; (3) from a single bag taken at random.

A sample taken from only one bag is manifestly unfair and inaccurate and the method hence should never be permitted. The separate testing of a number of samples, each taken from a single bag, involves usually a large amount of unnecessary work, especially if the lot represents a shipment of not more than 150 barrels. When a shipment of about 1,000 barrels is received the method may be employed, but even then it is preferable to subdivide the shipment and test an average sample from each portion. Occasionally, however, the testing of such samples may be desirable, especially with a new brand, as a check on its uniformity. The inspectors of the cement intended for use in the New York Rapid Transit Subway make their tests at the mill on eleven samples taken from each bin, ten of which are from borings made at different parts of the bin while the eleventh is a mixture of the other ten. For the usual condition, however, of shipments received in lots of 150 barrels or less, a single sample representing the average of the material is sufficient.

The proper number of bags to sample necessarily depends largely on local conditions which cannot be formulated. The Committee of the American Society of Civil Engineers* recommends that "where conditions permit, one barrel in every ten be sampled," and since four bags equal a barrel, this is equivalent to sampling one bag in forty. Although this amount is representative of average practice for larger lots, it is entirely too little for the small lots frequently received for short sections of sewer and other similar small jobs that are common in municipal work, where the entire amount of cement used may be only 30 or 40 bags, and which according to this method would be represented by a sample from only one bag. The author's practice is to sample 5 bags for every 50 barrels or less in the shipment, which agrees with the Committee's recommendation for large lots, but never permits less than 5 bags to be sampled.

In selecting the packages to be sampled, care should be exercised that they are taken from different parts of the pile and so fairly represent the average of the shipment. If it is possible, the best time to take the samples is when the cars are being

*See Appendix A.

FIG. 17.—Sampling Auger.

unloaded at the store-house, every bag in thirty or forty being opened and sampled.

The size of the samples depends on the number of tests to be made; for the ordinary tests as given in any one of the standard specifications, the amount should be between 8 and 10 pounds.

The material taken from any one package should also be an average of its entire contents, since the cement on its exterior is more liable to influences operating to change its properties than that on the interior, and also on account of the separation of coarse and fine particles in cases where the package has been subjected to much jolting in transportation. In sampling a barrel, a hole should be made in one of the staves midway be-

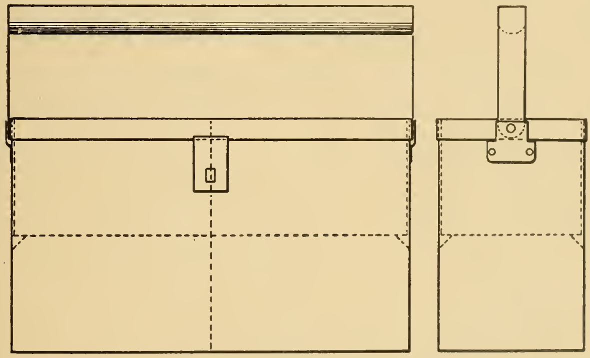


FIG 18.—Can for Collecting Samples.

tween the heads, and material removed from the center to the side. A sampling auger, or "sugar trier" (Fig. 17) is convenient for this purpose. A bag should also be sampled from surface to center, using either the auger or a long narrow scoop.

For collecting samples, the author uses the form of can shown in Fig. 18, which is divided into four compartments, the upper two of which are in a removable tray, the dimensions of the can being 14½ inches × 5 inches × 8 inches, and each compartment holding about 8 pounds of cement. Two of these cans are as much as a man can carry with comfort. When the samples are brought to the laboratory, they are emptied into a paper wash basin, thoroughly mixed with a spoon or trowel, then poured into sample cans similar to those in Fig. 19, except that they are provided with handles. The thorough mixing of a sample taken from several packages is a proceeding that never should be neglected. The laboratory of the Philadelphia Rapid Transit Co. uses square sample cans 2½ inches × 8 inches × 9 inches, and for collecting has a cover box holding four of these cans. This however makes an additional weight to carry and has the further disadvantage of not requiring the removal of the material on reaching the laboratory, thus encouraging the assistants to shirk the trouble of thoroughly mixing it. The square cans also are less easily handled than round ones although they take up less space in storage.

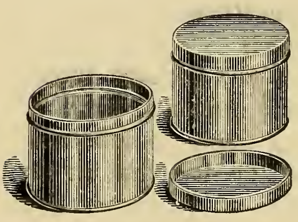


FIG. 19.—Sample Cans.

The only preliminary treatment required for the sample is in cases when it has become mixed with foreign matter such as sticks or stones or splinters from the barrel, or contains lumps, in which case it should be sifted through a coarse sieve, about 20-mesh, to remove them. This sifting has been recommended* for all samples to ensure a thorough mixing, but is not necessary nor as efficacious as a thorough mixing with a trowel or spoon.

After placing the cement in the sample can, it should be given a consecutive number, tightly covered and placed in a dry atmosphere at a normal temperature until tested. Samples collected in extremely hot or cold weather should always be brought to a temperature of about 70° Fahr. before testing.

*See Appendix A.

CHAPTER V.

THE TESTING OF CEMENT.

Tests of cement may be divided into two classes—research or experimental tests, and routine tests. Those of the first class are made for the purpose of determining how the material may be made and used to the best advantage and include such tests as investigations on the constitution of cement, determinations of its physical constants such as the coefficient of expansion and the modulus of elasticity, and also such tests as those of porosity, adhesion, effect of frost, effect of sea-water, and similar investigations which have as their object the study of the class of material as a whole. Routine tests are those whose purpose it is to determine whether a particular shipment of cement is of a quality sufficiently good for use in construction work. It is with this second class of tests that this book is chiefly concerned.

For a cement mortar or concrete to give good service in actual work it must possess two essential qualities—strength, or ability to carry the loads it is designed to sustain, and soundness, or ability to withstand any forces, either interior or exterior, which tend to destroy its permanency.

The routine or reception tests to which a cement is usually subjected are soundness, tensile strength (both neat and with sand), fineness, specific gravity, and time of setting. Other tests less frequently used are those of chemical analysis, compressive and transverse strength, apparent density, shearing and adhesive strength, microscopic examinations, etc.

These different tests may be classified as primary or secondary, the primary tests being those of strength and soundness, which qualities the material must necessarily possess, while the secondary tests, including those of specific gravity, fineness and chemical analysis, are those made to give additional information as to the ability of the material to qualify in the primary requisites, and have no intrinsic value in themselves. In the case of the fineness test, for example, no possible reason can be given for requiring the cement to be ground to a certain point, except that experience and investigations have shown that the fine material has greater strength and less liability to unsoundness, but otherwise the actual size of the particles can make no difference.

The determination of the time of setting can hardly be placed in either of these classes, for while it bears a relation to both soundness and strength, it is made chiefly for the purpose of ascertaining whether the material can be conveniently used, or that the time when it begins to harden will not be so soon that it cannot be properly placed, or so late that it will delay the progress of the work.

It has just been stated that the common test for strength is made in tension, while the compressive and transverse strength is determined but infrequently. The reasons for this will be given in some detail in Chapter IX., but it can be said here that although there is and can be no fixed relation between the strengths in tension, compression, cross breaking and shear, nevertheless the tensile strength is a comparatively fair measure of the ability of the material to withstand the other forms of stress, and, since it is by far the simplest of the strength tests to employ in routine testing, its adoption for this purpose is almost universal. The old argument that, since mortar and concrete are most frequently subjected to compression, the tests should also be so made, has been generally abandoned, and as a matter of fact, if the failures of cement constructions be studied, it will be noted that almost all of the failures of strength are due to weakness in tension, cross breaking or shear and rarely if ever to compression. In fact, the cracks usually to be observed in heavy monolithic concrete construction generally are due to temperature changes or shrinkage, the concrete actually failing in tension.

Development of Testing.—The development of cement testing runs more or less parallel to that of production. Smeaton's first tests were made by forming small balls of the material, placing them under water, and observing their hydraulic properties.

The first crude strength test was employed by Pasley, about the year 1830, and consisted in cementing bricks against a wall one at time, the second being cemented to the first and so on, the bricks forming a projecting beam, and the cement holding the greatest number of bricks being adjudged the superior. No distinction was made between quick and slow setting cements, and it is not even stated whether or not the bricks were added at fixed intervals. Pasley's next test was more scientific in its character and consisted of cementing together two bricks on end and determining the weight necessary to pull them apart. This appears to have been the origin of the tensile test. Pasley also determined the time necessary for a cement paste to harden under water. In the light of recent investigations on the subject of cement testing and in view of the great difficulties encountered by our scientific bodies in formulating accurate methods of testing, it is most interesting to note that Pasley as a result of his experiments endeavored "to lay down rules for judging the quality of cement offered for sale and for ascertaining whether it has been adulterated or not, by attending to which the most inexperienced person may easily detect such frauds in twenty-four hours or less," and also that "the comparative strength of cements may be judged of experimentally, and in a short space of time, such as ten days, with very little trouble, and the greatest accuracy."

Vicat, in 1828, devised a form of apparatus for determining the hardening of cement, consisting of a plunger loaded with a fixed weight which fell from a definite height into a cement paste, its penetration being a measure of the hardening. A modification of this apparatus, known as the Vicat needle, is the present standard for testing time of setting. The first cement briquettes made in France were molded in the form of a rectangle and when hard set were removed from the molds and notches cut in them to receive the clips.

John Grant, in 1858, when making tests of cement in connection with the construction of the London Main Drainage Works was the first to place them upon a scientific basis, and to develop definite methods. The form of briquette which he finally adopted after a long series of investigations is essentially the same as the English and American standards of today. His testing machine also was very similar in form to the long lever types now generally used.

Michaelis, in 1870, and Faija, in 1886, were the first to introduce forms of accelerated tests for soundness.

Methods of Testing.—The chief reason that it is difficult to secure uniform and accurate tests of cement lies in the fact that this material is one in which the tests, or at least the important ones, are made on artificially prepared specimens and not on the material in the form in which it is manufactured and sold. Cement is produced in the form of powder, tested for the most part as a paste, and used as a mortar or concrete. A specimen of iron, steel, wood or brick is shaped to fit the machine in which it is to be tested, but in so doing its physical properties remain unchanged, and the only errors that can be made are those due to incorrect manipulation of the machine. For the important cement tests, on the other hand, it is first necessary to prepare the specimens, the making of which completely changes the physical and chemical properties of the material, and which, therefore, must always be done in an accurate and uniform manner to produce true results.

Considered in this light, the ordinary tests of cement may be classified as either absolute or relative; the absolute tests being those of specific gravity, fineness and chemical anaylsis, which are made on the material as it is produced, while the relative tests include those of setting, strength and soundness, in which the material is changed both in form and in properties before the actual determination is made. It will be noticed that the essential tests of strength and soundness both come under the head of relative tests, and it is for this reason that the other tests are made, to give additional information as to the character of the material, for if strength and soundness could be determined absolutely there would be no necessity for any other tests.

Since the preparation of the specimens is so important a phase of the subject, it is especially necessary that uniform methods be followed if the results obtained by different laboratories and individuals are to be comparable. Recognizing this fact, committees of scientific societies have repeatedly attempted to formulate uniform methods of testing. The various committees of the German Association of Portland Cement Manufacturers, and the French Commission of 1891, on Standard Methods of Testing the Materials of Construction, have done notable work along these lines. In the United States, a Committee of the American Society of Civil Engineers, in 1885, produced the first standard for methods of cement testing. These rules, however, soon became obsolete so that the same body later appointed another Committee which submitted a Progress

Report,* in January, 1903, which, although by no means perfect, is a distinct advance on anything previously done for the promotion of uniformity in the methods of cement testing. Acting along similar lines, a Committee of the New York Section of the Society for Chemical Industry, in 1902, formulated a standard method† to be followed in the chemical analysis of cement.

Unfortunately, some of the most important operations in cement testing cannot be performed mechanically, and this furnishes one reason why it is such a difficult matter to formulate uniform methods, since in any process where personal equation is an important factor, practical experience is more essential than any amount of theoretical training or knowledge. In regard to this, the report of the Committee, of 1885, of the American Society of Civil Engineers says:

"The testing of cement is not so simple a process as it is sometimes thought to be. No small degree of experience is necessary before one can manipulate the materials so as to obtain even approximately accurate results.

"The first tests of inexperienced, though intelligent and careful persons, are usually very contradictory and inaccurate. and no amount of experience can eliminate the variations introduced by the personal equation of the most conscientious observers. Many things, apparently of minor importance, exert such a marked influence upon the results, that it is only by the greatest care in every particular, aided by experience and intelligence, that trustworthy tests can be made."

Requisites for Good Testing.—The prime requisites for the accurate and efficient testing of cement may be summarized as follows:

(1) The operators should be experienced and well-trained men, careful and conscientious.

(2) The various operations should be based upon a standard or pre-determined method and no deviations should be tolerated under any pretext.

(3) The methods should aim at the greatest accuracy and the greatest simplicity combined with an expenditure of the least amount of time and labor.

(4) Personal equation should be eliminated as far as possible from all operations.

(5) The records should be as complete as possible, but not unnecessarily complex.

*See Appendix A.
†See Appendix B.

CHAPTER VI.

SPECIFIC GRAVITY.

Definition.—The specific gravity of a substance is the ratio of the weight of that substance to the weight of an equal volume of water. Since, in the metric system, the cubic centimeter of water is taken as the basis of the gram weight, it follows that the specific gravity of a substance becomes the ratio of its weight in grams to its volume in cubic centimeters. This determination, therefore, consists of a measurement of weight and a measurement of volume.

Underburning.—The specific gravity of a well-burned cement is known to have certain definite limits. The higher the temperature used in burning, the more thoroughly will the ingredients be combined, thus giving by their contraction in volume a greater density or specific gravity. An underburned cement contains a large proportion of uncombined or insufficiently combined elements, some of which are sources of great danger, and in use may be sufficient to cause the disintegration of the cement, and the failure of the structure. Overburning, on the other hand, tends to break up some of the compounds which should be present in a normal cement, and to form other compounds that, although not generally injurious, are nevertheless possessed of much more feeble hydraulic properties, and thus tend to weaken the material.

It is thus evident that a normal cement must have been burned within a certain small range of temperature, and as the specific gravity is a measure of the degree of burning, it follows that if the cement be normal its specific gravity must lie within definite limits.

Adulteration.—Another important function of this test is the frequent detection of adulterants. Excluding plaster of Paris, or gypsum, the use of which is legitimate, the most common of the adulterants of Portland cement are raw-rock, slag, cinder, and natural cement, all of which have a much lower specific gravity, ranging from about 2.55 to 2.95. If a cement be of good quality it is frequently possible to add to it twenty,

twenty-five, or even a greater percentage of these materials, and, if thoroughly mixed, to have this addition escape detection in all of the physical tests with the exception of specific gravity, in which test the substitution at once becomes apparent. For example, if a cement (sp. gr. 3.15) be mixed with 10% of raw-rock (sp. gr. 2.64), the specific gravity will be lowered to 3.10, and with 25% will be reduced to 3.02, so that an addition of even 10% will be apparent to an operator familiar with the normal properties of that particular brand.

The specific gravity test alone, however, should never be relied upon for the detection of adulterants, since many other causes also may operate to produce an abnormally low value, chief of which are the age and the composition of the material,

TABLE VII.—Effect of Age on Specific Gravity of Cement.
(From Butler's "Portland Cement.")

	No. 1	No. 2	No. 3	No. 4
Specific Gravity when received	3 160	3.175	3.160	3.120
" " in 1 month	3.055	3.125	3 130	3.109
" " " 3 months	3.095	2.965	3.084	2.985
" " " 6 "	3 016	2.930	3 018	2.995
" " " 9 "	2.969	2.915	3.015	2.985
Loss in six months (per cent)	4 55	7 71	4 49	4.006

and in a lesser degree the fineness of grinding and the exterior conditions under which the test is made.

Effect of Age.—Cement exposed to the air gradually absorbs water and carbonic acid which, whether existing in a combined or in an absorbed state, materially tend to lower its specific gravity.

Table VII. is taken from a paper read by Henry Faija before the Society of Engineers, showing the results of experiments made to demonstrate this action. Unfortunately the conditions under which these tests were made are not given, but assuming them to be normal the falling off in specific gravity is unusually great. Such an extreme case as that of No. 2 in which the specific gravity falls off 0.210 in three months could occur but very infrequently. Also the fact that in two cases an increase is shown points either to peculiar conditions or inaccuracy in the work. The humidity of the atmosphere, of course, introduces a variable, but that could hardly operate to the extent of 0.04 as given for sample No. 1. The trend of the valves, however, is unmistakable.

Table VIII. shows the results of a similar series of tests made by the author. The cement in this case was a rotary Portland cement from the Lehigh Valley district, and was exposed to the air of the laboratory. These tests also show the same tendency although in a lesser degree.

Another series of tests, taken from the report of the Watertown Arsenal for 1901, is given in Table IX. This table also shows the effect of drying and igniting.

Drying a sample of cement at a temperature of 212° Fahr. has the effect of driving off the absorbed water while igniting

TABLE VIII.—Effect of Age on Specific Gravity.
(Tests by the Author.)

Age	Specific Gravity	Age	Specific Gravity
Original	3.134	1 Year	3.020
1 Month	3.125	1½ "	3.006
2 Months	3.121	2 "	3.000
3 "	3.109	2½ "	2.995
4 "	3 092		
6 "	3.073		
9 "	3.039		

TABLE IX.—Effect of Age on Specific Gravity of Cement.
(From Watertown Arsenal Report, 1901.)

BRAND	Specific Gravity				
	Original	After 14 days	After 28 days	After heating to 110° C.	After heating to redness
A	3.12	3.09	3.08	3.07	3.16
B	3.13	3.06	3 04	3.04	3.15
C	3.13	3.09	3 09	3.09	3.23

it with a blast lamp will restore it to the condition of the original clinker. This, however, is not true after a lapse of considerable time, since the water absorbed gradually attacks the cement and breaks it up into hydrated compounds, which when dehydrated have a lower density than that of the original material. A certain amount of storage is necessary for any cement, during which time the unstable and expansive elements absorb water and carbonic acid and thus become inert. A prolonged storage, however, produces the same action on the cement itself, until eventually it loses a great part of its hydraulic properties.

Effect of Composition.—The chemical composition of a cement affects its specfiic gravity both directly and indirectly, directly in that an excess of the heavier elements such as iron tends to increase its specific gravity, while the lighter elements tend to lower it, and indirectly in that the necessary degree of burning depends upon its composition. The temperature required for burning increases as the proportion of lime increases, and decreases as the iron and alumina increase. Two cements, therefore, may be equally well proportioned and burned and be subjected to the same conditions, and yet give very different results in the specific gravity, due to the difference in their chemical composition. For this reason the test must be considered a comparative and not an absolute one, so that the result of every determination must be compared with the normal value for that particular brand and not only with regard to a certain minimum specification.

TABLE X.—Effect of Fineness on Specific Gravity of Cement.
(Tests by the Author.)

	Fineness			Specific Gravity
	No. 50	No. 100	No. 200	
Original Cement	1.8	12.0	27.6	3.159
Reground once	0.0	4.0	11.3	3.164
" twice	0.0	0.0	0.6	3.166

TABLE XI.—The Specific Gravity of Different Sized Particles of the Same Cement.
(From Watertown Arsenal Report, 1901.)

BRAND	SPECIFIC GRAVITY				
	Size of Grain (inches)				
	>.0058	.0050	.0034	.0027	<.0027
A	3.09	....	3.12	3 12	3.04
B	3 07	3.12	3 08	3 09	2.99
C	3 08	....	....	3 00	3 02

Effect of Fineness.—The fineness of a cement affects the specific gravity in a slight degree, in that the coarse particles of clinker contain a small amount of air which cannot be eliminated by any process. The extent of this effect may be seen in Table X., which gives the results of tests made by the author, each value being the average of ten determinations. The fineness of the cement also affects the value in another manner, in that the finer the material the greater will be the absorption of water, hence the specific gravity of the finer particles will always be lower than that of the coarse. This is illustrated in Table XI., taken from the tables in the Report of the Watertown Arsenal for 1901. These two conditions tend to neutralize each other and for routine work need not be considered.

Effect of Humidity.—The humidity of the atmosphere introduces a small variable from day to day which must not be overlooked, although its amount is practically negligible. This is caused by the absorption of water by the cement on damp days, and the reverse condition in dry weather. This, of course, can be entirely eliminated by drying the samples. For all but the finest experimental work, however, this slight variable need not be considered.

In the routine testing of the specific gravity of cement, therefore, the operator has two conditions to investigate—degree of burning, and adulteration—and two variables to consider—amount of seasoning, and composition.

The Weight Test.—The original test to determine the amount of burning a cement received was the weight or the apparent density test, which consisted in mechanically filling a measure with cement, striking it off and determining its weight. This, of course, amounts to an extremely crude test of specific gravity, in which the measurement of volume is not made on the actual particles, but on the space loosely filled by those particles. The chief source of error in this method lies in the fact that the results are dependent so entirely on the uniform filling of the measure, the slightest jarring or irregularity in the process causing great errors in the results. The values also are dependent in a great degree on the fineness of the material, a coarser cement packing much closer, and hence weighing more than one which is fine and floury. Hence to require a high weight in this test is equivalent to asking for as coarse a material as will pass the actual specifications for fineness.

Four of the most common forms of apparatus used for this purpose are: (1) The double plane apparatus of the French Commission on Methods of Testing the Materials of Construction (Fig. 20), consisting of two planes at angles of 45°, the cement being placed on the end of the short plane, whence it runs through an opening at the bottom to the second plane and thence into the measure. (2) The apparatus designed by Prof. Tetmajer (Fig. 21), in which the cement passes through an oscillating sieve. (3) Faija's apparatus (Fig. 22), in which

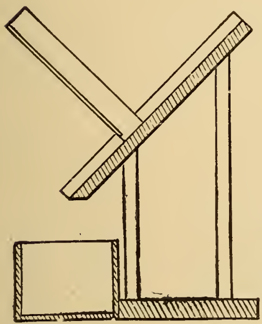


FIG. 20.—Apparent Density Apparatus Recommended by the French Commission.

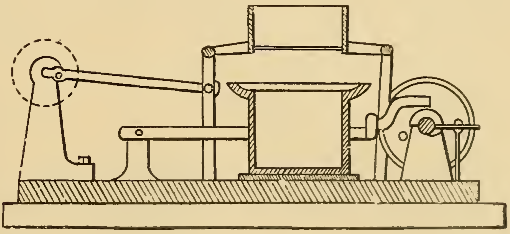


FIG. 21.—Tetmajer's Apparent Density Apparatus.

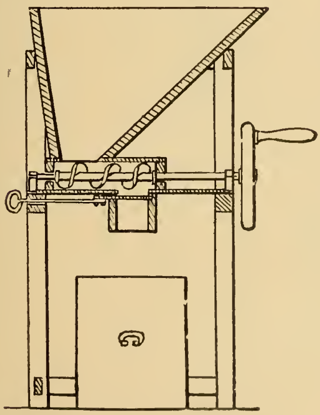


FIG. 22.—Faija's Apparent Density Apparatus.

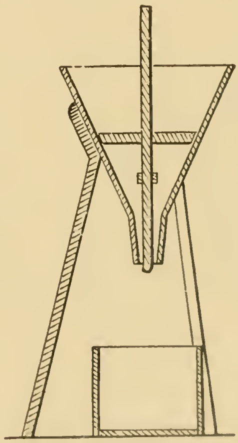


FIG. 23.—German Funnel Apparatus for Determining Apparent Density.

the cement is passed through a screw conveyor; and (4) the German funnel apparatus (Fig. 23), having in the center a rod which is rotated to facilitate and equalize the flow of material.

Several other forms of apparatus are used, but are generally similar in type to those shown. This determination is used extensively in European laboratories, but has never found favor in the United States, and is seldom, if ever, required.

Forms of Apparatus.—For making tests of the actual specific gravity of cement many forms of apparatus have been devised, all of which, however, are based upon the principle of measuring the amount of liquid displaced by a definite weight of material. Any liquid can be used provided it has no action on

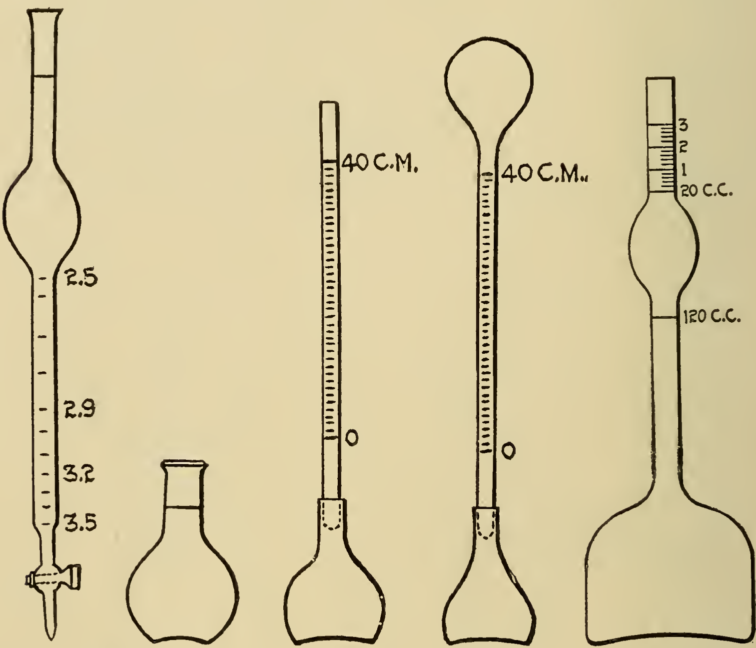


FIG 24. FIG. 25. Fig. 26. FIG. 27.

Forms of Apparatus Used for Determinations of Specific Gravity.

the cement, the most common being benzine, kerosene, turpentine or paraffine. Care must be taken that, whatever the liquid, it be free from water, and also that it be as little volatile as possible.

Mann's Apparatus.—Figure 24 shows the apparatus of Dr. Mann, consisting of a small flask which, when filled to a mark on its neck, contains a definite amount of the liquid. A burette graduated from the bottom and reading to one-tenth of a cubic centimeter is filled to a point equal to the volume of the flask. A definite quantity of cement is weighed and placed in the flask, which is then filled to the neck with liquid from the burette, the amount of liquid remaining in the burette after this operation being equal to the volume of the cement. As shown in the figure, the graduations may be so arranged that the reading can be taken directly.

The chief objection to this apparatus is the difficulty of removing the air held by the cement. By shaking the flask when a little over half full this error is partially but never wholly overcome. Also the shape of the flask, and the necessity for frequently handling it make the liquid more than usually susceptible both to evaporation and to changes of temperature.

Mr. Daniel D. Jackson, in the Engineering Record, recently described a method and apparatus similar to this in all essential particulars, and claimed for it an accuracy exceeding the Le Chatelier form. He, however, was compelled always to make a correction for temperature.

Schumann's Apparatus.—The Schumann apparatus (Fig. 25), consists of a long graduated tube having a funnel at one end, and the other end fitting closely into a flask of about 150 c. c. capacity, the tube being graduated from a mark near the bottom up to 40 c. c., in one-tenth c. c. divisions. The flask and tube are filled with the liquid up to the lower mark on the tube. A given quantity of cement, usually 100 grams, is poured slowly through the funnel, the elevation of the liquid giving the displaced volume.

This apparatus is open to several objections—the difficulty of introducing the cement without its adhering to the sides of the tube, the difficulty of eliminating air bubbles and the awkwardness of form. The cement can be prevented from adhering to the sides of the tube by using a long funnel, but since as much or even more material will stick to the funnel than to the tube, it is necessary to add enough material to elevate the liquid to a definite height, and to determine its amount by weighing the apparatus before and after its introduction. The size of the apparatus, moreover, makes the weighing difficult and awkward. With the exception of the

Le Chatelier, this form of apparatus, in spite of the many objections to it, is probably the most generally used.

Chandlot's Apparatus.—Chandlot's modification of the Schumann apparatus is shown in Fig. 26, the modification consisting in replacing the funnel end of the tube with a closed bulb. A mark is placed just below the bulb, so that when the bulb is inverted and filled with the liquid the amount contained will just equal that of the flask when filled to the lower mark. In using this apparatus 100 grams of cement are placed in the flask, the inverted bulb filled with benzine, and the connection carefully made with the tube in a nearly horizontal position. The liquid is then allowed to flow into the flask, the apparatus vigorously shaken to remove air bubbles, and the reading taken as with the Schumann apparatus.

The objections to this form are similar to those previously given; the impossibility of entirely removing the air by the rough shaking given, and the necessity of handling the apparatus, introducing probable errors through changes of temperature.

Le Chatelier's Apparatus.—Figure 27 shows the specific gravity bottle designed by Le Chatelier. The lower bulb with the tube above it contains 120 c. c. The bulb half way up the tube contains exactly 20 c. c., the 120 c. c. mark being placed directly under the bulb and the 20 c. c. mark above it. The tube above the upper mark is graduated into 1-10 c. c., starting from the upper mark and containing about 4 c. c., thus giving a capacity of 24 c. c. from the mark below the bulb to the top of the tube. The apparatus is about 30 centimeters in height, and the tube about eleven millimeters in diameter.

Two methods of using this apparatus were originally proposed: (1) The flask was filled with liquid up to the 120 c. c. mark. 64 grams of cement were then weighed out and gradually introduced into the end of the tube by means of a funnel until the liquid rose to the 20 c. c. mark. The remaining cement was then weighed and subtracted from 64, thus giving the amount in the flask, which quantity divided by 20 gave the specific gravity. (2) The lower part of the flask was filled with the liquid as before. 64 grams of cement were then weighed and the entire quantity introduced into the tube, making the liquid rise into the graduated portion. The reading on the tube plus 20 gave the volume displaced by the cement, 64 divided by this quantity giving the specific gravity.

The French Commission on Standard Methods of Testing, in proposing this form of apparatus for standard use, recommended a combination of these two methods, first employing method (1) as given, and then introducing into the flask the cement remaining after weighing, thus following the second method. The result of the test was to be the average of the two values thus obtained.

There are few objections to this form of apparatus, the air bubbles being freed from the material in its slow passage down the tube, the bulb preventing the cement from sticking to the sides, and there being no necessity for handling it.

This apparatus is certainly used to a greater extent in the United States than any other form, and has been recommended by the Cement Committee of the American Society of Civil Engineers, so that it may be considered the standard for making this test.

The Picnometer.—In cases where the specific gravity of cement is only determined at infrequent intervals, the picnometer or specific gravity bottle is often employed. This consists of a small flask usually of 100 c. c. capacity, and provided with either a mark on its stem, or a close fitting glass stopper having a capillary tube in its center. In making determinations with this bottle, it is first weighed empty, and then again when filled with benzine. The benzine is then emptied out and a weighed quantity of cement introduced, after which the flask is filled about two-thirds full with benzine and shaken vigorously to remove the air bubbles. It is then entirely filled and weighed. This weight less that of the cement and of the bottle gives the weight of the benzine in the flask, and if this quantity be subtracted from the weight of the benzine necessary to fill the flask, it will give the weight of the benzine displaced by the cement. Hence if this last weight be divided into that of the cement it gives the specific gravity of the cement relative to that of benzine, and if this quantity be then multiplied by the previously determined specific gravity of the benzine it will give the specific gravity of the cement. Expressed in a formula this becomes:

$$\text{Sp. Gr.} = \frac{C \times S}{C + B - W}$$

in which

C = weight of cement.
B = weight of bottle filled with benzine.
W = weight of bottle filled with cement and benzine.
S = specific gravity of the benzine.

The specific gravity of the benzine can be found either with an ordinary hydrometer or by use of the bottle itself. When the bottle is used determinations are made of the weight of the bottle empty, when filled with water, and when filled with benzine. The specific gravity of the benzine evidently then being:

$$\text{Sp. Gr.} = \frac{B - b}{H - b}$$

where

b = weight of bottle empty.
B = weight of bottle filled with benzine.
H = weight of bottle filled with water.

The specific gravity bottle is open to all the objections of presence of air, temperature changes, etc., that have been mentioned in reference to some of the preceding forms. To all but the skilled chemist accustomed to handling such apparatus, this method is necessarily crude.

Preliminary Treatment of Sample.—Since the object of this test is not only to determine whether the cement is underburned or adulterated, but also to find out in a measure the degree of seasoning received, it is recommended that the tests in routine work be made on the samples in the condition they are received and not on dried or ignited samples, although in every case where a cement falls below the specifications a second test should be made on a dried sample to ascertain whether an excessive seasoning has caused the low value.

Also for the reasons given on page 49, it is wrong to use cement that has been sifted for these tests, since there is considerable difference between the specific gravity of the coarse and fine particles.

Sources of Error.—The sources of error that are most likely to lead to erroneous results, and those most liable to escape detection are three:—(1) presence of air, (2) changes in temperature, (3) evaporation of the liquid.

The error due to the presence of air bubbles in the liquid can only be overcome by the exercise of considerable care. It is almost impossible to avoid this error in any form of apparatus in which the liquid is poured on the cement, but in those forms of apparatus in which the cement is introduced into the liquid, the air can be almost entirely eliminated if the operation be performed slowly, and if the receptacle be given an occasional slight jar to free any bubbles that may have found their way to the bottom.

Changes of temperature probably cause the majority of the errors made in specific gravity determinations. The actual temperature at which the test is made does not affect the results, but any change in that temperature at once introduces errors. For this reason the different forms of apparatus are often immersed in water before the initial and final readings are taken for a sufficient time to acquire the temperature of the water. This proceeding, however, requires considerable time, and is not necessary if care be taken that the room in which the tests are made is kept free from draughts of either hot or cold air, and that no other condition exists which is likely to cause changes in the temperature. Care especially must be taken never to touch the apparatus with the fingers, since their heat will appreciably affect the reading in a very short time.

The error due to evaporation is never great in the ordinary forms of apparatus if only a reasonable amount of time be employed in making the test. At ordinary temperatures, and where the entire determination can be completed in about five minutes, the evaporation may be entirely neglected. It should, however, be considered where a much longer time is required. In placing the apparatus in water to make the temperature correction, the top should always be tightly corked, since otherwise the error caused by evaporation may actually exceed the error that the operator is endeavoring to eliminate. Both the errors of temperature and evaporation can be detected by the use of a second flask, used as a blank, in which no cement is put, but which is subjected to the same conditions as the first, and on which readings are made before and after the experiment, the difference being the correction to be applied.

Another precaution that should always be employed is, after filling a flask, to allow a short time before taking the reading so as to permit the liquid on the sides of the tube to run down, this often making a considerable difference in the results.

The Author's Method.—The following method is used by the author in the Philadelphia Laboratories for making determinations of specific gravity:

All the samples which are to be tested, the apparatus, and

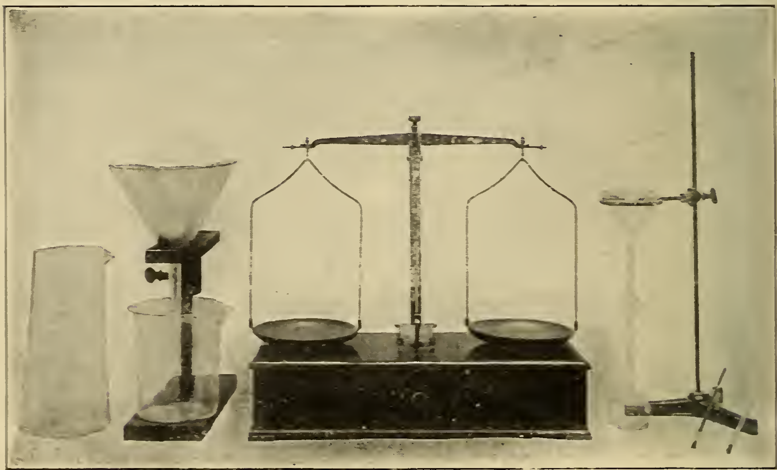


Fig. 28.—Apparatus Used by the Author for Making Determinations of Specific Gravity.

the benzine are first allowed to stand in the room in which the tests are to be made for at least an hour in order that they all may acquire a uniform temperature, care being taken that the doors and windows are so arranged that there can be no draughts nor currents of air. Two Le Chatelier specific gravity bottles, which have been carefully calibrated, are used alternately in making the determinations, and benzine, which has been determined to be neither very volatile nor hygroscopic, is employed for the liquid. Sixty-four grams of cement are weighed, on a piece of paper, in a beam balance of 5 milligrams sensibility (see Fig. 28). The flasks are filled with benzine a little above the lower mark, allowed to stand half a minute, and then adjusted carefully to the mark by means of a glass tube used as a pipette. A funnel, four inches in diameter, having a tube of such a width that it will just enter the top of the apparatus, is placed in the ring of a retort stand at such a height that the bottom of the funnel reaches about half an inch below the top of the flask, and also so that the apparatus is free to be moved up and down slightly without disturbing the funnel. The entire sixty-four grams of cement are poured into the funnel, the last traces of material being removed from the paper by means of a camel's hair brush.

The cement is then gradually forced through the funnel with a narrow glass rod, and at the same time the flask is given a jarring motion by raising it about a sixteenth of an inch and dropping it, holding it at the top of the tube where the temperature of the fingers will not affect the benzine. Working on a wooden table, and exercising reasonable care, there is no danger of breaking the apparatus, but if it is desired to further insure its safety, a piece of blotting paper may be placed under it. This jarring serves the double purpose of preventing the cement from clogging in the upper bulb, and also of freeing it from particles of air.

When all the cement has been introduced, the last traces are removed from the funnel and rod with the camel's hair brush, and the funnel removed. If any cement is clinging to the sides of the apparatus, it is pushed down with the glass rod, which is in turn scraped against the edge of the tube to remove the last traces of benzine.

After allowing the flask to stand half a minute, the reading is taken, interpolating to 1-100 c. c. A mark is placed on a far wall at the same height as the upper mark of the apparatus, so that by sighting on this mark, parallax is avoided. By means of a table similar to Table XII., the specific gravity is at once obtained and entered on the record. Since great care is exercised to prevent changes in temperature the flasks are not immersed in water during the operation.

The flasks are cleaned by inverting and shaking them over a precipitating jar about five inches in diameter, partially filling again and shaking until clean. The benzine is separated by

TABLE XII.—Values of Specific Gravity in Terms of the Readings of the Le Chatelier Apparatus, When Using 64 Grams of Cement.

	0.00	0.01	0.02	0.03	0.04	0.05	0.06	0.07	0.08	0.09
20.00	3.200	3.198	3.197	3.195	3.194	3.192	3.190	3.188	3.187	3.186
.10	3.184	3.182	3.181	3.179	3.178	3.176	3.174	3.173	3.171	3.170
.20	3.168	3.167	3.165	3.164	3.162	3.161	3.159	3.158	3.156	3.155
.30	3.153	3.151	3.150	3.148	3.147	3.145	3.143	3.142	3.140	3.139
.40	3.137	3.136	3.134	3.133	3.131	3.130	3.128	3.127	3.125	3.124
.50	3.122	3.121	3.119	3.118	3.116	3.115	3.113	3.112	3.110	3.109
.60	3.107	3.106	3.104	3.103	3.101	3.100	3.098	3.097	3.095	3.094
.70	3.092	3.091	3.089	3.088	3.086	3.085	3.083	3.082	3.080	3.079
.80	3.077	3.076	3.074	3.073	3.071	3.070	3.068	3.067	3.065	3.064
.90	3.062	3.061	3.059	3.058	3.056	3.055	3.054	3.052	3.051	3.049
21.00	3.048	3.047	3.045	3.044	3.042	3.041	3.039	3.038	3.036	3.035
.10	3.033	3.032	3.030	3.029	3.027	3.026	3.025	3.023	3.022	3.020
.20	3.019	3.018	3.016	3.015	3.013	3.012	3.011	3.009	3.008	3.006
.30	3.005	3.004	3.002	3.001	3.000	2.999	2.997	2.996	2.995	2.993
.40	2.992	2.991	2.989	2.988	2.986	2.985	2.983	2.982	2.980	2.979
.50	2.977	2.976	2.974	2.973	2.971	2.970	2.969	2.967	2.966	2.964
.60	2.963	2.962	2.960	2.959	2.957	2.956	2.955	2.953	2.952	2.950
.70	2.949	2.948	2.946	2.945	2.944	2.943	2.942	2.940	2.939	2.937
.80	2.936	2.935	2.933	2.932	2.930	2.929	2.928	2.926	2.925	2.923
.90	2.922	2.921	2.919	2.918	2.917	2.916	2.914	2.913	2.912	2.910
22.00	2.909	2.908	2.906	2.905	2.904	2.903	2.902	2.900	2.899	2.897
.10	2.896	2.895	2.893	2.892	2.891	2.890	2.888	2.887	2.886	2.884
.20	2.883	2.882	2.880	2.879	2.878	2.877	2.875	2.874	2.873	2.871
.30	2.870	2.869	2.867	2.866	2.865	2.864	2.862	2.861	2.860	2.858
.40	2.857	2.856	2.854	2.853	2.852	2.851	2.849	2.848	2.847	2.845
.50	2.844	2.843	2.842	2.841	2.839	2.838	2.837	2.836	2.834	2.833
.60	2.832	2.830	2.829	2.828	2.827	2.826	2.824	2.823	2.822	2.821
.70	2.819	2.818	2.817	2.815	2.814	2.813	2.812	2.811	2.809	2.808
.80	2.807	2.806	2.805	2.804	2.803	2.802	2.800	2.799	2.798	2.796
.90	2.795	2.794	2.793	2.791	2.790	2.789	2.788	2.787	2.785	2.784
23.00	2.783	2.782	2.781	2.779	2.778	2.777	2.776	2.775	2.773	2.772
.10	2.771	2.770	2.768	2.767	2.766	2.765	2.763	2.762	2.761	2.759
.20	3.758	2.757	2.756	2.755	2.754	2.753	2.751	2.750	2.749	2.748
.30	2.747	2.746	2.745	2.743	2.742	2.741	2.740	2.739	2.737	2.736
.40	2.735	2.734	2.733	2.731	2.730	2.729	2.728	2.727	2.725	2.724
.50	2.723	2.722	2.721	2.720	2.719	2.718	2.716	2.715	2.714	2.713
.60	2.712	2.711	2.710	2.709	2.708	2.707	2.705	2.704	2.703	2.702
.70	2.701	2.700	2.699	2.698	2.697	2.696	2.694	2.693	2.692	2.691
.80	2.690	2.689	2.688	2.686	2.685	2.684	2.683	2.682	2.680	2.679
.90	2.678	2.677	2.676	2.675	2.674	2.673	2.671	2.670	2.669	2.668
24.00	2.667	2.666	2.665	2.664	2.663	2.662	2.660	2.659	2.658	2.657

filtering, so that it can be used repeatedly, one gallon sufficing for about 100 determinations.

Two men make these tests, one weighing the cement and cleaning the flasks, and the other operating the flask and taking the readings. Experienced men following this method can easily make from 10 to 12 determinations in an hour. Two men in the Philadelphia Laboratories once made 54 tests in four hours, and some of these tests being repeated for curiosity it was found that in no case did the error exceed 0.006.

Whenever a test falls much below the average value for that brand of cement, or whenever it falls below specifications (3.100) a check test is made, and also a test made on a dried sample, all of which are entered in the records.

Determinations made by experienced men following this method can be considered accurate to the second decimal. The following are the results of ten tests on the same sample of cement made under the author's direction: These samples were mixed in with the regular tests of the day, and the operators had no knowledge that any special tests were being made, and hence exercised no unusual precautions. The results were: 3.143, 3.139, 3.141, 3.138, 3.138, 3.137, 3.136, 3.140, 3.143, 3.137; average, 3.1392; total range, 0.007; average departure from mean, 0.002; probable error of one result, 0.0017. It is thus evident that the statement that this method is accurate to the second decimal is well within the bounds of probability.

Interpretation of Results.—With the exception of the test for soundness, there is probably no test in which incorrect or misleading inferences can be drawn so readily as in that of specific gravity. This is chiefly due to the fact that the test is comparative rather than absolute. For example, one cement may average a specific gravity of 3.16 and another 3.11. Now, a sample of the first testing 3.12 might be underburned or considerably adulterated, and yet be higher than a normal value of the second brand. For this reason an operator must have considerable experience with average values before he can accurately interpret the result of a single test. Again, an abnormally low value may be the result of excessive seasoning. Now, if this seasoning has not been sufficient to lower the strength below requirements, the cement is undoubtedly in far better condition for service than if it were fresh, because it will almost certainly be volume constant. The greatest difficulty that many consumers experience is in the securing of well seasoned material, and yet testers of cement frequently reject such material merely because its specific gravity is low, thus defeating their own ends.

A sample of cement that tests below the average in specific gravity should be examined for adulterations and should also have tests made on dried and ignited samples, but if it is shown to be free from adulterations, sufficiently strong, and sound, it should never be rejected merely because its specific gravity may be somewhat below requirements.

The specific gravity clause, however, should be a feature of every specification, in order that a cement proven to be underburned or adulterated may be rejected on its strength, even though it may pass the other tests.

CHAPTER VII.

FINENESS.

Although the fineness to which a cement is ground may be of little consequence in itself, yet its effects on the other properties are so far reaching that the test becomes one of considerable importance. It is interesting to note how this condition has gradually become realized with the growth of the industry. In the old days of testing many of the records show cements leaving residues of as much as 25 or 30 per cent. on a No. 50 sieve, while a test leaving that much residue on a No. 200 sieve is now considered poor, some of the modern cements leaving a residue of even less than 15% on this sieve.

It has been definitely proven that, at least in the early stages of the hardening of cement, only the very fine particles are actively hydraulic. The fineness of the material, therefore, is a measure of its cementing value, and a fine cement, accordingly, will be much the stronger when mixed in a mortar, or it can be mixed with a larger proportion of sand than a coarse one and yet attain the same strength. For reasons explained later, however, a neat cement mixture is usually less strong when made of fine material, although this is comparatively unimportant on account of the infrequency of the use of neat mixtures in practice.

Also, since the hardening of cement is caused by the solution and subsequent crystallization of certain of its elements, it is evident that this action will be quickened by the fineness of the particles, hence the finer the grinding, the sooner will the ultimate strength be attained. Another effect of somewhat doubtful advantage is that the finer cement will be quicker setting, the same reasons given for early hardening applying to the setting with equal force.

Most important of all, however, is the fact that with finer grinding, the liability to unsoundness becomes less, since the small particles become seasoned more quickly and the expansive elements thus become inert.

Detailed discussions of the effect of fineness on time of setting, strength and soundness will be found in the chapters devoted to those subjects, but sufficient is here stated to show the reasons for and the importance of making this test.

Methods of Determining Fineness.—Fineness is customarily determined by sifting through a series of sieves of different mesh. Many other devices have been proposed, most of them attempting separation by currents of air or of a liquid, but none has yet proved sufficiently accurate and at the same time simple and quick enough to replace sifting in routine work. Separation by some such method is much to be desired, however, for the reason that even the finest sieve now in use is not capable of determining the size of the smallest, and hence the most active particles, and moreover, the amount of material passing the finest sieve does not necessarily give a measure of the amount of flour present.

"Dr. W. Michaelis,* the great German specialist, recom-

TABLE XIII.—Gradation of Fineness Recommended by Michaelis for Cement Testing.

(From Johnson's "Materials of Construction.)

Number of Meshes per Square Centimeter	Square Inch	Diameter of Wire In Millimeters	In Inches	Width of Mesh In Millimeters	In Inches	Area of Mesh In Square Millimeters	In Square Inches
900	4,200	0.133	0 0052	0.20	0.0080	0.04	0 0000610
3,600	23,500	0.067	0.0026	0.10	0.0040	0.01	0.0000150
15,000	97,000	0.033	0.0013	0.05	0 0020	0.0025	0 0000040
60,000	390,000	0.002	0 00008	0.02	0.0008	0 0004	0.0000006

mends† that two sieves be used, No. 75 and No. 150 (30 and 60 meshes per cm.) and in addition to these the Schöne washing apparatus with rates of upward flow of the alcohol of 2.8 inches per minute, giving particles of cement which would pass a No. 300 sieve (120 per centimeter), and also of 1 inch per minute upward velocity, giving particles which would correspond to those passing a No. 600 sieve (240 meshes per centimeter). This washing process added to the use of the two sieves would enable one to graduate the cement as in Table XIII.

"The relation between the largest diameter of particle and the rate of upward flow for absolute alcohol and Portland cement he finds to be

$$d = 0.036 \, v^{7/11}$$

where d = largest diameter in millimeters, and v = upward

*From Johnson's "Materials of Construction," pp. 412-413.
†"Thonindustrie-Zeitung," Aug. 24 and Nov. 23, 1895.

velocity of flow in millimeters per second in the cylindrical part of the washing apparatus."

"As a result of this further analysis for fineness, it appears that the conclusions drawn from an analysis with the No. 75 and the No. 175 sieves (30 and 70 per centimeter) may be entirely erroneous. Thus among the many analyses given by Michaelis in these articles are the two analyses in Table XIV. of cement ground in the same manner, on French buhrstones, 5 feet in diameter."

TABLE XIV.—Comparative Analyses of Two Cements.
(From Johnson's "Materials of Construction.")

Sieve-Gauges (Meshes per Linear Inch) Where Diameter of Wire = Width of Mesh	Sample No. 1 Parts	Sample No. 1 Total Passing	Sample No. 2 Parts	Sample No. 2 Total Passing
	Per Cent	Per Cent.	Per Cent.	Per Cent
Retained on No. 75 Sieve	0 65	99.35	1 55	98.45
Passed No. 75 and Retained on No. 175 Sieve	7.75	91.60	7 40	91.05
" " 175 " " " " 300 "	42.98	48 62	19.74	71.31
" " 300 " " " " 600 "	17 75	30 87	25 27	46 04
" " 600 Sieve	30 87	....	46.04	....
	100.00	......	100.00	......

"The total percentage passing the No. 175 sieve was 91.60 for sample No. 1, and 91.05 for sample No. 2. This would appear to give No. 1 a slight advantage. There was stopped at the next stage, however, 43 per cent. of No. 1 and only 20 per cent. of No. 2, thus leaving only 48.62 per cent. of No. 1 to pass the 300 sieve, while of No. 2 there passed 71.31 per cent. Finally there was but 31 per cent. of No. 1 to pass the washing test which corresponded to a No. 600 sieve, while 46 per cent. of No. 2 passed this last test of fineness. It thus appears that sample No. 2 is much finer ground than No. 1, although this would not appear from the most severe sieve-test it is possible to make, it being impracticable to use any finer sieve than about 175 meshes per linear inch (70 per centimeter)."

"Dr. Michaelis strongly urges, therefore, that in all scientific and expert investigations of fineness the washing tests be employed."

Figure 29 shows a common form of washing apparatus for separating a powder into different sized particles. A liquid under a constant head flows into the small tube, rises into the large tube, and overflows at the side. Before the flow of the liquid commences the powder is inserted into the large tube and vigorously stirred so that it is all in suspension, then, when the liquid begins to rise in the tube, the smaller particles are carried off, while the larger remain in suspension or collect at the bottom, from whence they can be drawn off through the stop-cock, collected, and the percentage determined. The size of the particles carried off, depends, of course, on the velocity of the liquid, which is regulated by adjusting the height of the reservoir.

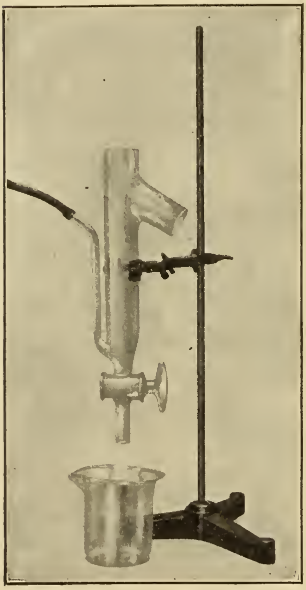


Fig. 29.—Apparatus for Determining Fineness by Washing.

Another method sometimes employed for crude washing tests of fineness consists in placing a definite amount of material in a beaker, filling it with water, stirring it, allowing it to settle for a few seconds, and then pouring off the water carrying the flour in suspension, repeating the process until the water is practically clear, drying the residue, and determining its amount. For rough tests, this method may be occasionally used, but for regular work it is crude and inaccurate.

In routine testing, therefore, recourse is made to sifting, not so much on account of any intrinsic merit in the method, but because no other scheme has been devised which is both adequate and practicable.

Sieves.—The sieves commonly found on the market for use in cement testing are the No. 50, No. 100 and No. 200. Sieves intermediate between these, especially the No. 74, No. 120 and No. 175 have been, and even still are occasionally employed, but scarcely to an extent to warrant consideration. Of these different sieves, the No. 200 is by far the most important, since only that part of the cement passing this sieve is truly active, at least in the early stages of its hardening. Moreover, it is not true, as many imagine, that any definite ratio exists between the residues on the No. 100 and No. 200 sieves. This is clearly shown in Table XV. The series of tests in this table are taken from regular routine determinations made in the Philadelphia Laboratories and show that the amount of residue on the No. 100 gives scarcely any indication of what will remain on the No. 200. It is, therefore, evident that the use of the No. 200 sieve is essential if the effective fineness of the cement is to be determined.

The employment of the No. 100 sieve is desirable since it shows the gradation of the material, and also since it marks in a measure the amount of material capable of possessing any cementing properties. Cement particles passing the No. 100 sieve always possess some activity, but those remaining on this sieve may, for all practical purposes, be considered inert.

TABLE XV.—Showing Lack of Proportionality Between Residues on No. 100 and No. 200 Sieves.

(Tests by the Author.)

No.	Fineness			Ratio of
	No. 50	No. 100	No. 200	No. 200 to No. 10.
1	0.0	4.8	21.9	4.6
2	0.1	8.2	17.7	2.2
3	0.0	6.4	25.3	4.0
4	0.0	2.1	18.0	8.6
5	0.2	10.4	25.0	2.4
6	0.1	6.3	19.2	3.0
7	0.0	14.7	27.3	1.9
8	0.5	5.8	19.2	3.3
9	0.0	3.6	18.0	5.0
10	0.0	9.2	24.1	2.6

The No. 50 sieve gives little additional information, but as the results are often interesting in showing the character of the clinker, and sometimes in detecting adulterants, and since the test requires but an insignificant amount of time, when made in connection with the other siftings, its use is comparatively general.

The intermediate sieves give little or no additional information, and hence are not recommended.

Wire Cloth.—Probably the greatest difficulty in the way of the standardization of the fineness test is encountered in the procuring of uniform wire cloth. At present, the greatest variation will be found even among the so-called standard sieves. No. 50 and No. 100 sieves of comparative accuracy

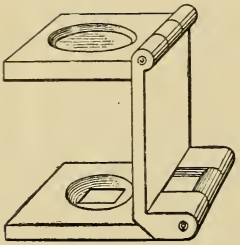


FIG. 30.—Linen Tester's Microscope.

can be procured, but even the best No. 200 sieves rarely count over 194. The author has seen, in laboratories of considerable reputation, No. 100 sieves counting as low as 78 × 84, while No. 200 sieves counting but 150 are by no means uncommon. Evidently, many of the seeming discrepancies in the results obtained by different laboratories can be traced to this source alone. Standardization of wire cloth can probably be reached best by stipulating definite limits by actual count of the number

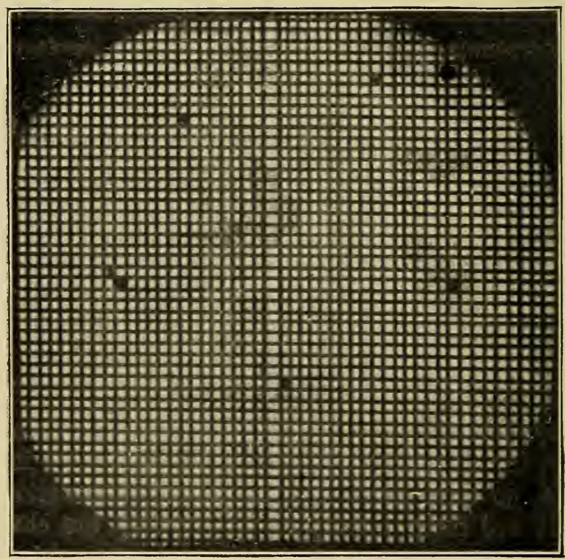


FIG. 31.—Illustration of Defective Wire Cloth.

of meshes per inch. Two other methods, often adopted, are testing by comparison with a standard, and finding the diameter of the largest particles passing the cloth. The first of these methods, however, has the objection of removing a basis of comparison between different laboratories, while the second is rather too difficult for the average cement tester.* The simplest method of counting wire cloth is by the use of an ordinary

*For a description of this method see article by Allen Hazen in the report of the Massachusetts State Board of Health for 1892.

linen tester's microscope (see Fig. 30), which can be procured at an insignificant cost, is sufficiently accurate, and furnishes a method of standardization that can be readily employed even by the poorest equipped of field laboratories. Of course, it is evident that a piece of wire cloth might count exactly the proper number, and yet be far from accurate. This is well shown in the photographs* in Figs. 31 and 32. Both of these

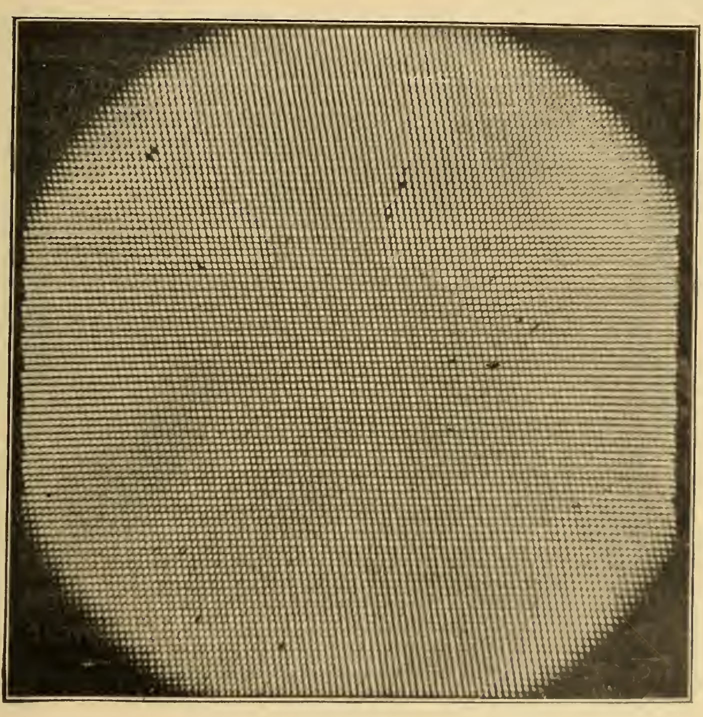


FIG. 32 —Illustration of Defective Wire Cloth.

sieves show irregularity in spacing, while the finer sieve also shows distortion of mesh probably caused in mounting. Distorted sieves or those as badly spaced as that in figure 31, should never be used, but if the only irregularity consists of one wide space it may, if desired, be stopped with solder.

In regard to the diameter of the wire from which the sieves are made there is comparative uniformity, nearly all the makers using the following sizes:—for No. 50. 35 O. E. gauge.

*Photographs loaned by Henry S. Spackman Engineering Co., Phila., Pa.

for No. 100, 40 O. E. gauge, and for No. 200, 42½ B. and S. gauge. It has frequently been urged that, in order to obtain better uniformity, the wire in all sieves be made one-half the width of the opening. This suggestion, however, is scarcely practicable, since none of the standard gauges in the market conforms to the diameter necessary to meet this requirement. Moreover, the wire for the No. 200 sieve would be only 0.0017 inches in diameter, which would be difficult, if not impossible, to procure, and would at best make an extremely flimsy and easily broken sieve. The expense and difficulty of procuring this wire, therefore, would scarcely be warranted for the sole purpose of obtaining a uniform ratio between the width of the wire and the opening.

The following specifications* have been used by the author for the past four years, in purchasing wire cloth for use in the Philadelphia Laboratories, and have been proven satisfactory:

1. Cloth for cement sieves to be of brass wire of the following diameters:

No. 50, No. 35 O. E. gauge, 0.0090 inches.
No. 100, No. 40 O. E. gauge, 0.0045 inches.
No. 200, No. 42½ B. and S. gauge, 0.00235 inches.

2. Mesh to count as follows:

No. 50, not less than 48, nor more than 50 per linear inch.
No. 100, not less than 96, nor more than 100 per linear inch.
No. 200, not less than 188, nor more than 200 per linear inch.

3. Cloth for No. 50 and No. 100 sieves to be woven; cloth for No. 200 sieve to be twilled.†

4. Mesh to be square and to show no great irregularities of spacing.

Mechanical Sifting.—For the mechanical operation of sieves, a great number of devices, more or less ingenious and efficient, have been designed. Figure 33 shows a sifting machine formerly used in the Philadelphia Laboratories. This was described by Mr. R. L. Humphrey‡ as follows: "It is operated

*These specifications have recently been adopted by the Committee of the American Society of Civil Engineers on Uniform Tests of Cement. See Appendix A.

†Woven wire overlaps every alternate strand—twilled wire laps the strands in pairs. Woven cloth of 200 meshes cannot be procured in the market.

‡In a paper entitled "A Few Remarks on the Testing Laboratory of the City of Philadelphia," read before the Engineers' Club of Philadelphia, April 1, 1899.

by a small electric motor, and consists of a wooden frame (1 ft. 10 ins. long, 14 ins. wide and 10 ins. deep), supported by four legs; a box 14 ins. long, 10 ins. wide and deep, fits closely into the frame. This box has trunnions in the center of two sides which move in grooves in the outside frame; and is moved to and fro by a crank connected to a crank-disk having a throw of $1\frac{1}{2}$ ins. The box holds four sets of brass sieves, each set

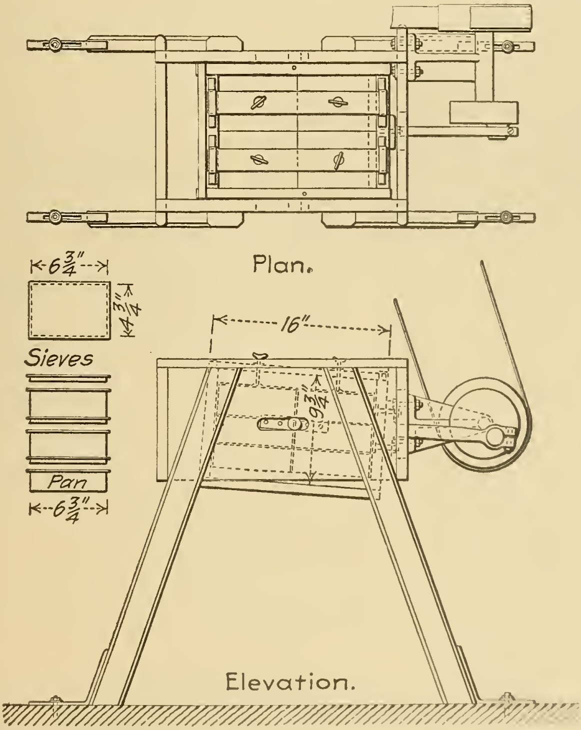


FIG. 33.—Mechanical Sifting Machine Used in the Philadelphia Testing Laboratories.

consisting of a No. 100 and a No. 200 sieve, a cover, and a pan, all nesting into each other. These sieves are held in place by means of two clamps. The driving pulley makes 100 revolutions per minute, and the box holding the sieves makes 200 movements per minute to and fro. The sieving frame has a tilting motion, and the rapidity of the motion imparts to it a jerking movement."

The Riehle hand sand sifter (Fig. 34), as its name implies, was designed for the sifting of sand, but may also be used for cement. The frame holding the sieves is fastened to an upright revolving crank at one end, while the other end is held in a slot in the upright. The turning of the hand wheel gives to the sieves a circular motion and also a jar at the point where the crank passes its dead center.

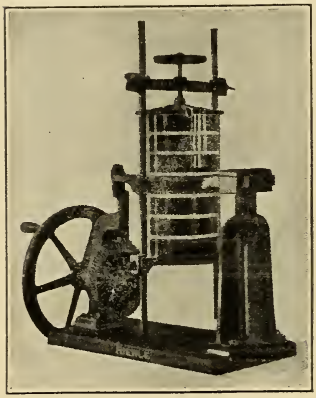


Fig. 34.—The Riehle Hand Sand Sifter.

A simple device for cement sifting is shown in Fig. 35. This apparatus was designed and is used by Mr. S. S. Voorhees, and its operation is self-explanatory. An ordinary fan motor furnishes the power, driving the frame through a crank wheel having a throw of 2 ins. The holes to receive the sieves are about half an inch larger than the sieves themselves, which are strapped in loosely to allow some play. A rubber mat is used to break the pound on the bottom of the sieve. The motion given to the sieves is the same as that of the machine in Fig. 34, except that in this case the throw is vertical, while the other is horizontal.

These three machines can be considered as types of the best of the many sifting devices in use, although the actual number of different forms nearly equals the number of testing laboratories.

Appliances for the mechanical sifting of cement are employed either for the purpose of securing greater uniformity and accuracy, or to economize time and labor. It has been the author's experience, however, that neither of these results is accomplished for the following reasons: In machine sifting one of two methods must be followed, (1) the machine is given a definite number of turns or shakes and the amount passing or retained on the sieves weighed directly, or (2) the sifting is continued until a definite amount of material ceases to pass the sieves at the end of a definite number of turns or shakes.

The first of these methods is manifestly unfair and inaccurate, because of the different speeds with which cements will sift under different conditions, those most affecting it being the age and specific gravity of the material, and the dampness of the material and of the atmosphere, conditions with which all practical operators are thoroughly familiar, but which are often overlooked by those directing the work. As an example of the effect of age on the speed of sifting, the author tested a fresh cement on a mechanical sifter and obtained a certain result at the end of thirteen minutes; four months later, it was again sifted under precisely similar conditions, and reached

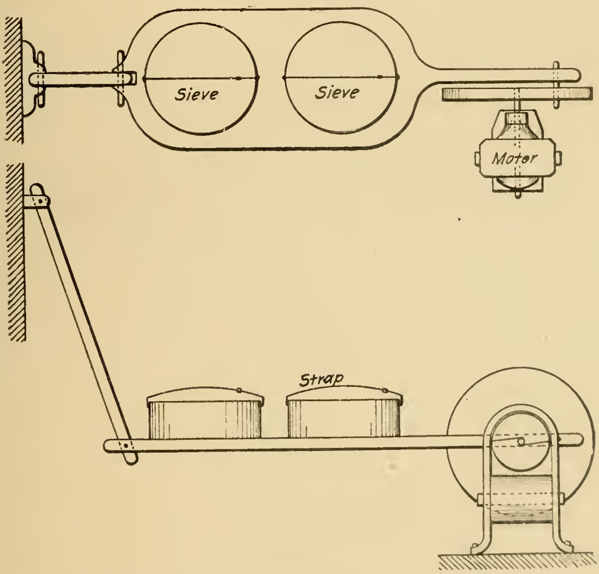


FIG. 35.—A Simple Design for a Mechanical Sifter.

the same result in seven and a half minutes, and at the end of thirteen minutes showed a fineness 2.4% greater than that previously obtained. The effect of the other conditions is obvious. Other things being equal the heavier cement will sift faster than the lighter one, while excessive dampness will tend to retard it. Two cements, therefore, of actually the same fineness may give widely variant results if tested in this manner.

The second of these methods is more accurate, but the attendant disadvantages are: First, that it introduces the personal equation, to eliminate which so much trouble has been taken, because the exact point of completion is left more or less to the discretion of the operator, and, second, that it actually takes more time to sift in this manner, where the sieves must be fitted together and put into the machine several times for each determination, than in the method of hand sifting, where the point of completion is more easily observed.

To the arguments against mechanical sifting must be added the cost of installing another piece of apparatus, and the slight likelihood of this method being adopted by the smaller laboratories, thus making comparisons of results difficult. Although devices for machine sifting, therefore, may be valuable in permanent laboratories for experimental work, they generally are neither economical nor more reliable in ordinary routine.

Size and Shape of Sieves.—Cement sieves are usually circular in shape, and 6 or 8 ins. in diameter. Those advocating the use of the larger sieve claim that the area of the smaller size is such that the time required for making a test is unnecessarily prolonged. On the other hand, those favoring the six-inch sieve claim that the larger sieve is more difficult to manipulate, is less likely to contain uniform cloth on account of its greater area, and also, unless its sides are very heavy, is more liable to become distorted in use, thus either breaking the cloth, or, by stretching, rendering it inaccurate. Since both of these claims have undoubtedly good foundation, the author has compromised on a seven-inch, which is found, in a large measure, to overcome the objections made to both the other sizes. Of course, this applies only to the No. 200 sieves; for the No. 50 and No. 100, six inches is amply sufficient. Although cement sieves almost universally are circular, it is, nevertheless, sometimes claimed that square sieves are preferable on account both of the greater ease of mounting the cloth on them squarely in the first place, and also because they are less liable to become distorted. If, however, care is taken when purchasing sieves to see that the cloth is properly mounted, and that the sides are sufficiently heavy, this objection becomes trivial.

Two and a half or three inches is a convenient depth for sieves, the cloth being placed half an inch from the bottom. The No. 200 should also be provided with a closely fitting pan about two inches deep.

Treatment and Size of Sample.—It is frequently recommended that samples should be dried* before testing for fineness, although the only advantage gained by this process is a slight increase in the actual speed of sifting, the results being practically unaffected. On the other hand, it involves the use of a drying oven, which takes time to operate, and money to install, and only adds one more item to the already too complicated program of cement testing, so that for routine work it is not advantageous. The only necessary preliminary treatment of the sample is when the cement is lumpy, or when foreign matter such as splinters from the barrel has become mixed with it, in either of which cases the material should be sifted through a coarse sieve, about 20-mesh, before being weighed.

Although 100 grams are generally taken for making the fineness test, most experienced operators find 50 grams amply sufficient. The time of sifting is practically halved by this reduction in the size of the sample, and at the same time the ac curacy obtained is all that is necessary.

Necessary Degree of Accuracy.—It is the practice of most of the manufacturers to report the results of the fineness test to the nearest per cent. The majority of Government and municipal laboratories report to a tenth per cent., while many carry their results to hundredths. In one case, the author saw the report of a small field laboratory in which the results were given to *three* places of decimals.

In this connection a test recently made by the author is of interest. Ten samples of cement were taken from ten different bags in the same car-load shipment, and presumably from the same bin at the works, and each sample was sifted separately to see what variation existed in the cement itself. The cement was a rotary Portland of high reputation. The siftings were made with extreme care and check tests made to insure the greatest accuracy possible. The results obtained are given in Table XVI., and show a range of 0.7% on the No. 100 sieve, and 2.3% on the No. 200. Is it not absurd to report results to hundredths or even tenths, when the cement itself on the fine sieve shows a variation of over 2%?

It will be found upon investigation, that even the most experienced operators rarely work with a probable error of less

*See Appendix A.

than one-half of one per cent., so that generally this may be taken as the limit of accuracy in tests of fineness by sifting. Placing the limit this low has also the advantage of making check tests and duplications more easy. It is very hard for any two operators to agree as to when the sifting is complete, if it is carried to tenths or hundredths, but the point where the residue cannot be reduced by more than a half per cent. is comparatively well marked.

Methods of Operation.—Although the manipulation of cement sieves appears to be comparatively simple, it is nevertheless surprising to find how many operators naturally take the inaccurate or the tedious method. If, however, the method is based upon the following considerations, it cannot go far wrong:

TABLE XVI.—Showing Variation in Fineness in Different Bags of Same Shipment. (Tests by the Author.)

No.	Fineness		
	No. 50	No. 100	No. 200
1	0.2	8.1	19.7
2	0.3	8.4	20.6
3	0.2	8.4	20.8
4	0.4	8.6	21.2
5	0.4	8.7	22.0
6	0.2	8.3	20.2
7	0.4	8.8	21.8
8	0.3	8.2	20.0
9	0.3	8.7	21.6
10	0.2	8.2	19.9
Variation	0.2	0.7	2.3

The residue on the sieves should always be weighed—not the amount passing through. No matter how carefully the test may be conducted, some of the fine powder is certain to escape, amounting in some instances to as much as one per cent. This lost powder is almost entirely that part which passes the No. 200 sieve, hence the reason for weighing the residues.

The method should be so arranged that the point of completion can be readily observed. This may be accomplished by sifting over a sheet of paper, so that the amount passing the sieve can be seen at once, or if a pan is used, it should be emptied frequently so that the progress of the sifting may be noted.

Sifting simultaneously through a nest of sieves is not advisable. In the first place, when nests are used, there is a tendency for the fine powder to drift back through the various sieves, thus making clean sifting very difficult. Again, the nest is much more difficult to operate that a single sieve. Also, it is a longer method, since the greater part of the sample must pass through all three sieves, whereas, in sifting separately, only the residue from one sieve is put through the next. Of course, when using the sieves separately, the operation should begin with the finest sieve. Notwithstanding the obvious diminution of labor obtained by sifting in this order, it is remarkable how many operators start with the No. 50, thus wasting considerable time and effort.

The use of shot or pebbles, on the sieves, to force the cement through more quickly, is permissible. Tests have frequently been made to ascertain whether the grinding action so produced is appreciable, and it has been found that in no case does it amount to more than a couple of tenths of a per cent., although their use often halves the time of manipulation.

Common Sources of Error.—The most common sources of error in determinations of fineness are: Flaws in the sieve. This can only be obviated by careful watching. The sieves should be carefully examined every day before making the tests, and those showing flaws should either be discarded, or, if the flaw be small, stopped with solder. A No. 200 sieve carefully used should last for 300 to 400 tests.

Loss of powder: This is always due to carelessness in manipulation. The operator making these tests should be a man sufficiently conscientious to repeat his work in case he spills an appreciable amount of the material.

Weighing amount passing sieves instead of residues: As previously stated this procedure will consistently lower the results from one-half to one per cent.

The Author's Method.—The sieves are circular, 2½ ins. in height; the No. 200 sieve is 7 ins. in diameter, while the No. 50 and No. 100 are 6 ins.; the pans are 2 ins. in depth. The wire cloth is made to conform with the specifications on page 70.

The cement, if lumpy, or if containing foreign matter, is first sifted through a No. 20 sieve, and any small lumps passing through are crushed. Fifty grams of cement are then carefully weighed on a balance sensible to about five centigrams (see Fig. 36*), and placed on a No. 200 sieve, with a pan attached. The operator grasps the sieve in one hand, so that it slopes upward toward the other hand, which gives it a rapid

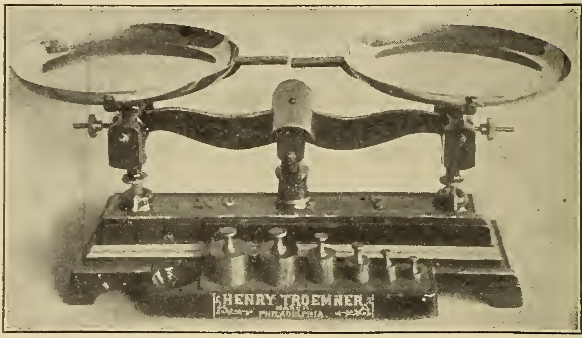


FIG. 36.—Scales for the Fineness Test.

succession of rather sharp blows; care must be taken to slant the sieve so that the cement is evenly distributed. It also must be struck squarely on the side, since hitting on the top or bottom tends to throw out the material. When most of the fine powder has passed the sieve, the pan is emptied, replaced, a few ounces of shot poured in and the sifting continued. This quickly forces the remainder of the powder through, and a point is soon reached, where it is impossible to reduce the residue by a half of a per cent. even after long sifting. The pan is then emptied and placed under a No. 20 sieve, through which the residue is poured, thus freeing it from the shot. The resi-

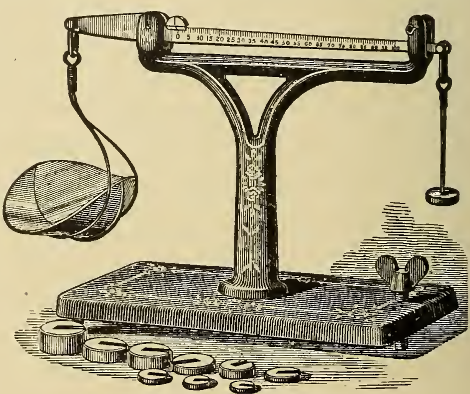


FIG. 37.—Special Scales for the Fineness Test.

*Figure 37 shows another form of balance especially designed for this test. Using 50 grams, the beam is so graduated that the residue or amount passing may be read directly.

due is then weighed and placed on the No. 100 sieve, where the operation is repeated. The determination for the No. 50 is made similarly, except that in this case it is not necessary to use shot. It is convenient, when using 50 grams, to have the weights of the balance marked double their real value, so that the amount of residue gives the percentage directly.

Sifting through the No. 200 sieve by this method takes on an average 5 or 6 minutes; through the No. 100 about a minute; while 3 or 4 shakes will be sufficient for the No. 50. The total time occupied in making a complete determination of fineness is thus about 8 or 10 minutes. With an experienced operator, the tests can be considered accurate to the nearest half per cent., although they are reported to the nearest two-tenths.

Interpretation of Results.—In considering the results of the fineness test, it must be remembered that fineness is not an end in itself, but only the means to an end, its purpose being to ensure the soundness and increase the strength of the material. Unless, therefore, the material is exceedingly coarse, it is generally unwise to reject a shipment on the fineness test alone, if it is otherwise satisfactory. The manufacturer should be notified, however, and if future shipments show no improvement that brand should be prohibited on the work.

CHAPTER VIII.

TIME OF SETTING.

Definitions.—When cement is mixed into a paste with water, and allowed to stand, it gradually loses its plasticity and begins to offer resistance to external forces. When this resistance is complete and the material is ruptured by an attempt to change its form it is said to have "set." The increase in strength afterwards acquired is known as "hardening." These two actions are more or less independent, there being no fixed relation between them.

From a physical standpoint, two stages in the setting may be recognized:—first, when the mass begins to offer resistance, and second, when this action is completed, or when the mass cannot be appreciably distorted without rupture. In practice, these stages are termed "initial set" and "hard set," and determinations of time of setting are made to ascertain the time required for a cement paste to reach these two critical points.

Objects of Test.—That a cement paste or mortar should set within definite limits of time is a practical consideration rather than one affecting its strength or permanence. In actual construction, a cement should not have begun to set before being placed in the work, since any subsequent working of the mortar, unless thoroughly retempered, tends to break up the crystals already formed, thus weakening it, and also rendering it more susceptible to disintegration. A partially set mortar also will not flow as easily into the voids of the aggregate thus making a poorer bond. On the other hand, after it has been placed in the structure, it should set and harden as quickly as possible so that it can offer a definite resistance to any external forces which might destroy it if still plastic. The best cements, therefore, should be slow in acquiring initial set, but after having reached that point, should become hard set quickly, and hence specifications usually give a cement a minimum time for acquiring initial set, and a maximum for hard set.

Theory of Setting.—The chemical processes involved in the setting and hardening of cement are not yet definitely known. "M. Fremy* considered Portland cement to be very complex in composition, and ascribed the setting to the action of lime upon certain puzzolanic compounds, composed of double silicates of lime and alumina, the calcination of the clay giving rise to a porous material which absorbs the lime by capillary affinity."

"M. Landrin concluded that a substance corresponding to the formula 3 SiO_2, 5 CaO is found in both Portland cements and puzzolana, and he considered this to be the active element in the hardening of cement, although he states that aluminate of lime contributes to the setting and accelerates that action."

"Prof. Le Chatelier, from his study of Portland cements, explains the phenomena of setting by showing that certain salts, including the aluminate and silicate of lime which form the active elements of Portland cement, while soluble in an anhydrous state, form insoluble salts when hydrated. When they come into contact with water in mixing mortar the anhydrous salt enters into solution, then, becoming hydrated, the hydrate is precipitated from the saturated solution in a crystalline form. Those salts which are thus capable of being dissolved in an anhydrous state and then becoming hydrated arrive at stability in two ways—by decomposition and by combination."

"The tricalcic silicate, which is the essential element of Portland cement, is decomposed in presence of water to a hydrated monocalcic silicate and a hydrate; thus

$$SiO_5Ca_2 + Aq = SiO_2, CaO, 2.5\ H_2O + 2\ CaO, H_2O."$$

"The monocalcic silicate crystallizes in the form of needle-like crystals and the hydrate in hexagonal lamina visible to the eye. The tricalcic aluminate is hydrated by simple combination with the water.

$$Al_2O_6Ca_3 + Aq = Al_2O_3, 3\ CaO, 12\ H_2O."$$

Mr. Clifford Richardson describes† the setting of cement as follows: "On the addition of water to a stable system made up of the solid solutions which compose Portland cement, a new component is introduced which immediately results in a lack of equilibrium, which is only brought about again by

*From Spalding's "Hydraulic Cement."

†"The Setting or Hydration of Portland Cement," a paper read before the Association of Portland Cement Manufacturers, Dec. 14, 1904.

the liberation of free lime. This free lime, the moment that it is liberated, is in solution in the water, but owing to the rapidity with which it is liberated from the aluminate, the water soon becomes supersaturated with calcic hydrate, and the latter crystallizes out in a network of crystals which binds the particles of undecomposed Portland cement together. From the characteristics of the silicates and aluminates it is evident that the latter are acted upon much more rapidly than the silicates, and it is to the crystallization of the lime from the aluminates that the first or initial set must be attributed. Subsequent hardening is due to the slower liberation of lime from the silicates. If the lime is liberated more rapidly than it is possible for it to crystallize out from the water, expansion ensues and the cement is not volume constant."

"The set of Portland cement is almost entirely due to the decomposition of the alit alone. Examination of a thin section of a neat Portland cement mortar will show that it contains large quantities of unattacked celit and a certain amount of unattacked alit."

"The strength of Portland cement after setting is due entirely to the crystallization of calcium hydrate under certain favorable conditions and not at all to the hydration of the silicates or the aluminates, since in this act of hydration nothing can take place which would tend to bind these silicates and aluminates together. Celit is certainly decomposed to but a slight degree in the process of setting. From this we may infer that the strongest cement is the one which contains the smallest amount of celit and such a conclusion is entirely justified by experience."

Effect of Composition.—Since the aluminates are chiefly active in the setting of cement, it follows naturally, other things being equal, that the higher the percentage of alumina present the quicker will be the setting. Since the majority of Portland cements run rather high in alumina, and since they are required to be very finely ground, which also accelerates the setting, under normal conditions they would set so quickly as to be entirely unfit for use. In order to retard the setting, it is now common practice to add one or two per cent. of gypsum, or plaster of Paris, to the finished cement. This admixture is generally made immediately before the final grinding, so that the mixture is a thorough one. The effect of this addition of sulphate of lime on the setting and strength of cement is of such importance that the following researches of M. Chandlot* are given at some length:

"Setting.—The retardation of set caused by the addition of gypsum to cement varies according to the quantity of gypsum employed" (see Table XVII.).

"But the action of gypsum on the setting of cement is not always permanent, and it very often happens that if a cement containing an admixture of gypsum is gauged a long while after the gypsum has been added, it sets very quickly; this effect is produced particularly with cements which set very

TABLE XVII.—Effect of Gypsum on Portland Cement.
(Tests by Chandlot)

Quantity of Gypsum Added	Set of the Neat Cement with Fresh Water											
	1				2				3			
	Initial Set		Hard Set		Initial Set		Hard Set		Initial Set		Hard Set	
Per Cent.	H.	M.	H.	M.	H.	M.	H.	M.	H.	M.	H.	M.
0.0	0	7	0	22	0	7	0	15	0	2	0	5
0.5	0	50	2	43	0	10	0	17	0	2	0	5
1.0	2	40	4	50	3	50	5	0	1	30	2	35
1.5	2	57	5	17	3	50	5	0	3	20	5	15
2.0	3	0	5	20	4	20	6	45	4	0	7	0
3.0	3	0	6	40	3	45	7	0	5	0	7	0
4.0	3	30	7	0	5	0	7	0	5	0	7	0

quickly when they have no admixture of gypsum, and which after the gypsum has been added, have been exposed to the air for several days" (see Table XVIII.).

"When a cement is preserved from contact with air, the set may again become quick after a lapse of a very long period. Table XIX. shows cement mixed with 2% of gypsum, and preserved in an air-tight flask."

"Cements containing an admixture of gypsum, present this peculiarity, that they often set more rapidly when gauged with sea water, than when gauged with fresh water."

"When a cement containing gypsum has been exposed to

*From "Ciments et Chaux Hydrauliques," by E Chandlot. Translation from Butler's "Portland Cement."

the air and has become quick setting again, it sets quicker when gauged to a thin consistency with an excess of water, than when it is gauged to a stiff paste with very little water. This

TABLE XVIII.—Effect of Gypsum on Portland Cement.
(Tests by Chandlot.)

Description of Cement	Set with Fresh Water — Initial Set		Hard Set	
	H.	M.	H.	M.
1. Cement Mixed with 3 Per Cent. of Gypsum—				
Tests made the same day....	1	0	7	0
" 4 days after....	0	5	2	15
" 7 "	0	5	0	20
" 11 "	0	8	0	30
" 15 "	0	5	0	30
" 19 "	0	7	0	35
" 24 "	0	5	0	25
" 32 "	0	10	0	30
" 41 "	0	45	5	30
2. Cement Mixed with 2 Per Cent. of Gypsum—				
Tests made the same day....	5	0	19	0
" 12 days after....	4	40	14	0
" 21 "	0	18	0	50
3. Cement Mixed with 1 Per Cent. of Gypsum—				
Tests made the same day....	5	30	8	30
" 8 days after....	0	18	2	30
" 15 "	0	11	0	20
4. Cement Mixed with 1 Per Cent. of Gypsum—				
Tests made the same day....	6	0	9	30
" 8 days after....	4	30	8	0
" 15 "	0	15	0	30
" 30 "	....		7	0

TABLE XIX.—Effect of Gypsum on Portland Cement.
(Tests by Chandlot.)

	Initial Set		Hard Set	
	H.	M.	H.	M.
Tests made the same day..........	3	0	6	25
" 1 month after...........	2	50	5	0
" 2 " "	1	30	7	0
" 5 " "	0	10	0	18

is contrary to that which always takes place with cements not containing admixtures of gypsum."

"Strength.—The addition of small quantities of gypsum to Portland cement results in increasing its strength. When, however, a cement is kept in sea water and the proportion of gypsum added exceeds one to two per cent., the mortar is not long before it shows traces of alteration, and the briquettes are sometimes completely disintegrated" (see Table XX.).

"If the cement containing gypsum is allowed to remain in sacks for several weeks, it develops very poor strains at the early dates of testing" (see Table XXI.).

"As a result of a considerable series of chemical researches in the matter, M. Chandlot comes to the conclusion that the peculiar effects of adding gypsum to Portland cement are due to the formation of a sulpho-aluminate of lime, a salt which he succeeded in producing artificially, and to which he attributes the formula (Al_2O_3, 3 CaO) 2.5 (SO_3, CaO). From this he deduces the following theory:

"It is well known that aluminate of lime is insoluble in a saturated solution of lime. If then, sulphate of lime and free

TABLE XXII.—Effect of Gypsum on Portland Cement. (Tests by Chandlot.)

Periods After Which Samples of the Liquid Were Taken.	Matters in Solution Per Litre of Liquid in Grammes.		
	CaO.	Al_2O_3.	SO_3 CaO.
	Gr.		Gr.
10 minutes	1.085	Nil	1 734
3 hours	1.085	"	1 632
6 "	0.875	"	1.632
12 "	0 930	"	1.504
8 days	1.085	"	Nil
1 month	0.304	"	"

lime are present, together with aluminate of lime, it follows that the combination of the sulphate of lime with the aluminate can take place but very slowly, because the aluminate cannot become hydrated on account of the immediate solution of the lime. Thus, a mixture of powdered aluminate of lime, of sulphate of lime and of slaked lime, having been shaken with an excess of distilled water, produced the results given in Table XXII."

"The above shows that the combination of the sulphate with the aluminate only takes place after a considerable period, the aluminate becoming hydrated but very slowly."

"In Portland cements of the best manufacture, there always exists a little free lime, and as they contain very little alumina, this free lime, by rapidly dissolving, prevents the hydration of the aluminate; the sulphate of lime becoming dissolved in

TABLE XX.—Effect of Gypsum on Portland Cement. (Tests by Chandlot.)

Tests with Fresh Cement.

Description of Mortar	Period Elapsed Since Gauging	Tensile Strength in Kilogrammes per Square Centimetre — Quantity of Gypsum Added to the Cement — 0 Per Cent.		1 Per Cent.		2 Per Cent.		3 Per Cent.		4 Per Cent.	
		Fresh Water	Sea Water	Fresh Water	Sea Water	Fresh Water	Sea Water	Fresh Water	Sea Water	Fresh Water	Sea Water
Neat Cement	7 Days	34.2	39.2	45.5	53.1	37.5	43.0	30.6	31.5	18.6	25.0
	28 "	47.4	51.6	51.9	60.6	47.5	58.7	55.7	63.4	34.0	26.2 (1)
	3 Months	51.7	54.5	55.1	50.7	59.0	65.9	62.1	69.3 (1)	65.2	25.6 (3)
	6 "	50.9	51.5	59.2	59.7	57.0	50.0 (1)	63.2	67.0 (3)	72.4	0 (4)
	12 "	60.6	25.1	59.7	48.3	63.9	31.9 (3)	65.6	72.0 (3)	80.0	0
Mortar 3 to 1	7 Days	15.7	14.9	17.7	17.0	18.5	16.2	13.0	13.4	8.9	9.9
	28 "	23.4	18.0	26.6	23.1	26.6	20.2	25.2	18.4	20.5	0 (4)
	3 Months	30.0	21.6	34.2	25.9	32.7	24.2	33.6	23.6	31.0	0
	6 "	32.7	23.1	37.7	25.7	35.7	28.0	38.2	29.2	36.1	0
	12 "	35.5	28.2	37.7	27.5	36.0	31.9	37.7	31.0	41.5	0
Mortar 3 to 1 Briquettes Kept in Air	7 Days	16.9	....	19.7	....	19.9	....	15.5	....	11.7	....
	28 "	28.6	....	35.9	....	33.7	....	29.5	....	26.9	....
	3 Months	35.2	....	43.4	....	42.9	....	40.1	....	42.2	....
	6 "	44.2	....	45.2	....	45.6	....	50.5	....	46.9	....
	12 "	53.0	....	49.5	....	50.4	....	51.1	....	58.2	....

NOTE:—1 Kilogramme per square centimetre = 14.228 lbs. per square inch.

(1) Briquettes showing some signs of alteration.
(3) Briquettes very much cracked and blown.
(4) Briquettes completely decomposed.

TABLE XXI.—Effect of Gypsum on Portland Cement. (Tests by Chandlot.)

Tests Made with the Same Cement Kept in Sacks for Two Months.

Description of Mortar	Period Elapsed Since Gauging	Tensile Strength in Kilogrammes per Square Centimetre									
		Quantity of Gypsum Added to the Cement									
		0 Per Cent.		1 Per Cent.		2 Per Cent.		3 Per Cent.		4 Per Cent.	
		Fresh Water	Sea Water	Fresh Water	Sea Water	Fresh Water	Sea Water	Fresh Water	Sea Water	Fresh Water	Sea Water
		K.	K.	K.	K.	K.	K.	K.	K.	K.	K.
Neat Cement	7 Days	28.0	35.6	28.5	34.1	21.0	25.2	15.0	13.4	11.6	12.6
	28 "	47.0	48.5	42.9	48.2	37.5	48.9	34.2	40.9	22.9	16.5 (2)
	3 Months	52.2	63.1	51.7	56.1	53.3	50.9	48.7	48.1 (1)	50.9	37.7 (3)
	6 "	55.2	49.2	54.9	55.5	49.2	52.0	51.2	52.0 (2)	58.6	43.4 (3)
	12 "	59.7	17.6	56.0	27.7	53.0	50.5	58.5	51.6 (2)	54.7	46.0 (3)
Mortar 3 to 1	7 Days	14.1	15.2	15.2	13.6	15.7	13.0	6.6	7.0	5.1	5.6
	28 "	23.7	22.1	22.0	22.2	21.5	18.0	21.6	18.0	17.2	12.1
	3 Months	29.7	27.0	29.2	26.2	28.2	20.9	33.7	25.0	28.5	19.5
	6 "	31.9	27.5	31.9	24.4	34.2	24.7	35.7	25.6	37.3	22.4
	12 "	35.6	29.4	36.1	27.5	34.4	26.5	38.0	25.6	36.5	26.2
Mortar 3 to 1 Briquettes Kept in Air	7 Days	13.7	....	19.1	....	15.7	....	6.2	....	5.1	....
	28 "	28.6	....	31.0	....	28.6	....	26.9	....	23.7	....
	3 Months	37.6	....	41.1	....	35.6	....	36.6	....	33.7	....
	6 "	47.2	....	49.9	....	43.7	....	47.5	....	43.0	....
	12 "	56.7	....	59.9	....	51.1	....	57.7	....	56.7	....

NOTE:—1 Kilogramme per square centimetre = 14.228 lbs. per square inch.

(1) Briquettes showing some signs of alteration.
(2) Briquettes cracked.
(3) Briquettes very much cracked and blown.

turn, and not being able to combine with the aluminate, adds its action to that of the lime in annulling the function of the aluminate; as it is to this salt that setting is attributable when it takes place rapidly, a slow-setting cement results."

"If the free lime, by being sufficiently exposed to the air, becomes carbonated, at the moment when the cement comes into contact with the water, the lime dissolves less freely, and nothing prevents the solution of the aluminate; the combination with the sulphate of lime can take place, and the sulpho-aluminate formed, as well as the excess of aluminate, by crystallizing, determine the rapid setting of the cement."

Effect of Age.—When cement is allowed to stand exposed to the air and to dampness, it gradually absorbs water and carbonic acid. This absorption and consequent hydration affects the aluminates more readily than the silicates, so that cement thus exposed gradually becomes slower setting, until eventually it loses all of its hydraulic properties, although well protected cement may be stored several years without appreciable deterioration. Generally speaking, however, it may be said that the tendency is for a cement to become slower setting with age, the only exception being in cases where a high percentage of sulphate of lime is present (see page 83).

Table XXIII. gives the results of a series of tests made by the author to demonstrate this action. The cement was a high grade rotary Portland, and was kept in its original package in the air of the laboratory, all tests being made at a uniform temperature.

Effect of Mixing Water.—Since the setting of cement is caused by a solution and subsequent crystallization of certain of its ingredients, it follows that the greater the amount of water present, the longer the time it will take for it to reach the saturated condition necessary for crystallization.. The effect of different percentages of water on the setting may be clearly seen in Tables XXIV. and XXV.

The temperature of the water also affects the setting, increased temperatures accelerating the rate of crystallization. Spalding* says: "Below a certain inferior limit, ordinarily from 30 to 40 degrees Fahr., the mortar sets with extreme slowness or not at all; while at a certain upper limit, in some

*"Hydraulic Cement," by F. P. Spalding, p. 65.

TABLE XXIII.—Effect of Age on Time of Setting.
(Tests by the Author.)

Age of Cement	Time of Setting (in minutes) Initial	Hard	Age of Cement	Time of Setting (in minutes) Initial	Hard
Original	32	179	1 year	182	420
1 month	35	173	1½ years	209	510
2 months......	41	226	2 "	267	600+
3 "	61	240	2½ "	303	600+
4 "	92	265			
6 "	106	330			
9 "	135	358			

TABLE XXIV.—Influence of Amount of Mixing Water on Time of Setting.
(From Sabin's "Cement and Concrete.")

	Per Cent. Water, by Weight.	24	26	28	30	32	34	36
Portland Cement	Time of Setting (minutes)							
	Initial Set..........	2	2	3	7	21	28	38
	Hard Set...........	160	188	279	289	371	403	583
	Per Cent. Water, by Weight.	26.7	28.6	30.8	33.3	36.4	40.0	....
Natural Cement	Time of Setting (minutes)							
	Initial Set...........	20	23	30	42	46	55	..
	Hard Set...........	28	41	57	76	78	85	..

TABLE XXV.—Showing Effect of Amount of Mixing Water on Time of Setting, and Also a Comparison of the Gillmore and Vicat Needles.
(From Watertown Arsenal Report, 1901.)

Brand	Per Cent. of Water	Time of Setting: With Gillmore Wires, Initial H.	M.	Hard H.	M.	With Vicat Needle, Initial H.	M.	Hard H.	M.
A.	20	2	20	5	00	0	35	4	25
	25	3	20	7	30	2	50	6	35
	30	5	40	..	..	4	40	8	40
B.	20	4	05	7	10	2	45	6	10
	25	5	10	8	05	3	35	7	05
	30	7	00	..	..	5	30	..	.
C. (with plaster)	20	2	10	4	25	0	50	3	00
	25	4	35	6	00	3	00	5	30
	30	5	45	..	..	5	10	7	15
C. (without plaster)	20	0	05	0	15	0	05	0	10
	25	0	35	4	55	0	10	3	30
	30	5	10	8	35	3	15	6	50
D.	20	1	40	5	10	1	28	4	44
	25	4	15	6	05	3	25	5	40
	30	4	50	7	10	4	33	6	53
E.	20	0	30	4	35	0	05	3	40
	25	4	10	6	40	3	10	6	10
	30	5	35	8	05	4	50	7	20

cements between 100 and 140 degrees Fahr., a change is suddenly made from a very rapid to a very slow rate, which then gradually decreases as the temperature increases, until practically the mortar will not set." For the small ranges ordinarily occurring in routine testing, however, say between 65 and 75 degrees Fahr., the effect is usually negligible.

Effect of Fineness.—It is obvious that a finely ground material will be more quickly attacked by a solvent than a coarse one, so that a fine cement is almost invariably quick setting unless artificially retarded, even in cements thus treated the finer being generally the quicker setting. Table XXVI. gives the

TABLE XXVI.—Influence of Fineness on the Time of Setting of Portland Cement.

(From Butler's "Portland Cement.")

Sample No.	How Treated	Fineness Residue on No. 50	No. 76	No. 180	Time of Setting in minutes Initial Set	Hard Set
1.	As received	0.0	6.0	22.4	25	45
	Reground	0 0	0.0	Tr.	1	5
2.	As received	2.7	10.0	26.6	30	90
	Reground	0.0	0.0	Tr.	6	15
3.	As received	1.5	7.6	24.4	30	120
	Reground	0.0	0.0	Tr.	7	15
4.	As received	1.2	6.7	30.0	20	60
	Reground	0.0	0.0	Tr.	2	10
5.	As received	1.0	9.3	28.4	15	30
	Reground	0.0	0.0	0.6	1	10
6.	As received	0.5	7.7	26.4	Undefinable	360
	Reground	0.0	0.0	0.4	8	25
7.	As received	0.8	3.0	18.0	15	240
	Reground	0.0	0.0	0.4	2	240
8.	As received	3.6	16.0	34.8	20	30
	Reground	0.0	0.0	1.6	2	5

results of tests showing the effect of regrinding cements on the time of setting. The acceleration of the setting in this case, however, is not entirely dependent on the fineness, but also on the seasoning, since the interior of the coarse particles contains fresh material practically unseasoned, while the original fine material has passed the early stage of rapid setting. To determine the effect of fineness alone, a clinker should be ground, at the same time, to different degrees of fineness, and the time of setting determined, in which case the variable introduced by seasoning would be eliminated.

Effect of Exterior Conditions.—Of the different exterior conditions affecting the setting of cement, temperature and humidity are by far the most important. Increased temperature tends to accelerate the crystallization and make the setting rapid, while increased humidity tends to retard it. High temperatures generally quicken the rate of setting whether acting before or during the test, so that if a sample of cement is exposed to an excessive heat its setting is usually accelerated, even if it be cooled to normal temperature before the determination is made. For this reason, shipments of cement made in summer will often be slow setting when leaving the manufacturers and quick when arriving on the work, the heat in the cars having brought about this condition. The temperature of the laboratory exercises the same influence, as can be

TABLE XXVII.—Influence of Temperature on the Setting of Portland Cement. (From "Butler's Portland Cement.")

Sample No.	Temperature Fahrenheit — Initial Set in Minutes				Temperature Fahrenheit — Hard Set in Hours			
	100°	80°	60°	40°	100°	80°	60°	40°
1.	1.5	4	6	13	1.25	1.5	2	2.5
2.	3	5	6	8	1	1.25	1.75	2.5
3.	4	10	15	20	0.5	0.75	1.5	6.5
4.*	5	9	15	30	0.5	0.75	1	6
5.	6	10	14	25	1	1.5	2	2.5
6.*	7	12	15	20	1.75	2	2.25	2.5
7.*	9	10	15	17	3.5	6	7	12
8.	10	15	35	40	0.75	1	1.25	1.75
9.	11	15	20	57	3	5	6	10
10.	11	13	15	30	2.5	3	3.5	6
11.	19	32	60	120	3	6	7	15
12.	15	35	70	360	3.5	6	7	22

* Adulterated with Kentish Rag-stone.

seen from Table XXVII. The effect of dampness on cement prior to the test, as has been shown, is to season it and thus retard its set. The same action also is apparent during the tests, those made in a damp atmosphere being much slower than those made in dry air. Test specimens stored in a damp closet, accordingly, will set slower than if kept in dry air.

Rise of Temperature.*—The rise of temperature in cement pastes during setting has been the subject of frequent controversy, some engineers having gone to the length of stating that the only necessary specification to ensure a good material

*For comprehensive data on the rise in temperature of mortars and concretes, the reader is referred to the Report of the Watertown Arsenal, "Tests of Metals, etc.," for 1901.

was that it should show no appreciable temperature rise in setting. The reason for this was that the rise in temperature was supposed to be a direct measure of the so-called "free lime" in the cement. As a matter of fact, however, it is due more to the heat of crystallization of the normal ingredients, and its amount will generally be found to be a function of the time of setting. If, therefore, a cement is so made that it will develop no heat, it must, of necessity, be very slow setting, and to be brought to this condition, the majority of cements would require the addition of an excessive amount of sulphate of lime.

It has been suggested that the time of setting be measured by temperature rise, the time taken to reach the maximum being called initial set, and the return to the normal, hard set. This, however, has been proven impracticable, both by reason of the variable introduced by the presence of free or loosely combined lime, and also by reason of the very small rise occurring in slow setting cements, which would make readings of time very difficult, if not impossible. The author determined this rise of temperature on every sample of cement tested in the Philadelphia Laboratories for over four years, and at the end of that time abandoned the test as not only inconclusive, but also as often actually misleading.

The rise in temperature of a paste of normal cement, in a mould the size of the ring used with the Vicat needle, will be found to average from 3 to 5 degrees Fahr., and also since its amount varies more or less with the time of setting, the effect of age, temperature, fineness, etc., will be similar.

Normal Consistency.—Since the amount of water used in mixing exerts considerable influence on the time of setting, it follows that this percentage must be definitely fixed and not left to the discretion of the operator. Some specifications, notably those of the U. S. Army Engineers, require that all cements be mixed with the same amount of water, but on account of the varying composition and other characteristics of different brands the consistency of the pastes so obtained will be quite variable, and since it has been determined that the action of different cements is more nearly similar when gauged to a definite consistency, rather than with a fixed amount of water, the plasticity or the consistency of the paste should always be uniform. This "normal consistency" can be obtained by several methods, the three following being those most generally employed:

The Ball Method.—The consistency obtained by this method is such that if a ball of the paste, about two inches in diameter, be dropped upon a hard surface from a height of two feet, it will not crack nor flatten to more than half its original thickness. This determination is extremely simple and easy to make, gives a consistency readily distinguished and suitable for moulding into any form, and for an experienced operator, is accurate to one-half of one per cent.

By the Vicat Needle.—(See page 94.) This method requires a consistency such that a cylindrical plunger, one centimeter in diameter and of 300 grams weight shall penetrate a definite distance into the paste contained in the rubber ring. The French Commission on Methods of Testing, recommended that this penetration be 34 millimeters; the Committee of the American Society of Civil Engineers advises 10 millimeters, while a consistency corresponding with that obtained by the ball method will be found to be about 7 or 8 millimeters. Although this method is somewhat more accurate than the ball method, it is much more tedious, usually requiring several trials before the proper consistency is obtained, and is often impossible for quick setting cements.

The Fluid Method.—This method originated in the Royal Testing Station of Charlottenburg and consists of mixing the cement to a syrupy paste so that it will run from the blade of a spatula ($6 \times 1\frac{1}{2}$ ins.) in long thin threads without forming lumps. Representing the amount of water required to bring the paste to this consistency by N, then the percentage to be used for neat pastes is $\frac{N + 1}{2}$, and for 1 : 3 sand $\frac{N + 3}{4}$. So far as the writer's experience has gone, however, this method is not as accurate as either of the two preceding, there being even greater room for difference of opinion as to when the cement will just run off the knife without forming lumps than as to when the ball or the plunger behaves properly. This method in common with the Vicat has the disadvantage of requiring a separate determination, whereas in following the ball

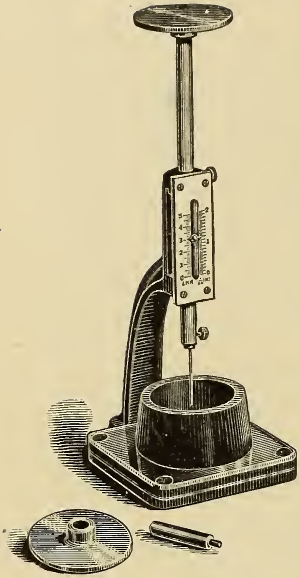


Fig. 38.—The Vicat Needle.

method the consistency determination can be made in connection with the other tests.

Consequently, in routine work, the ball method is generally preferred as being quick, convenient and sufficiently accurate. If it is desired to use the consistency recommended by the Committee of the American Society of Civil Engineers, that of the ball method plus one per cent. will be found near enough for all practical purposes.

Forms of Apparatus.—But two forms of apparatus are used in the United States for routine determinations of time of setting—the Vicat and the Gillmore needles.

The Vicat needle, shown in Figure 38, consists of a stand supporting a needle one millimeter in diameter, and loaded to 300 grams weight. The cement paste is placed in a rubber mould in the shape of a frustrum of a cone having an upper diameter of 6 centimeters, a lower diameter of 7 centimeters and 4 centimeters in height, the mould resting on a glass plate an eighth of an inch thick. The specimen is placed under the needle which is let down upon it from time to time, the amount of penetration being read from the graduated scale. Initial set is said to have taken place when the needle ceases to penetrate within five millimeters of the bottom of the specimen, and hard set is attained when the needle ceases to indent its surface. For making the normal consistency test, previously described, the plunger is substituted for the needle, the difference in weight being compensated by changing the upper cap.

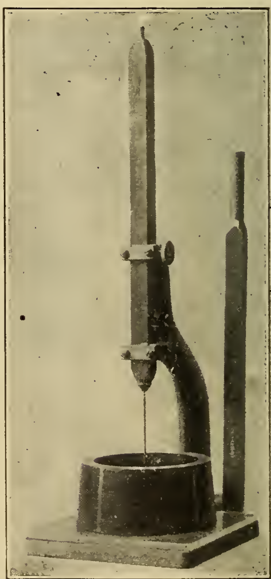


Fig. 39.—An Improved Form of Vicat Needle.

An improved form of Vicat needle, made by Jos. W. Bramwell, is shown in Fig. 39. The movable part, consisting of a square rod having the needle fastened to its lower extremity, weighs exactly 300 grams and is graduated to indicate the amount of penetration, while for determinations of consistency, a second rod, terminated by the plunger, may be substituted for that carrying the needle, or, if it is desired, a single rod may be obtained, having the needle at one end and the plunger at the other. This form of apparatus eliminates the possibility of error due to the use of the wrong cap, and also commends itself not only for its simplicity of construction and ease in manipulation but also by its lower cost.

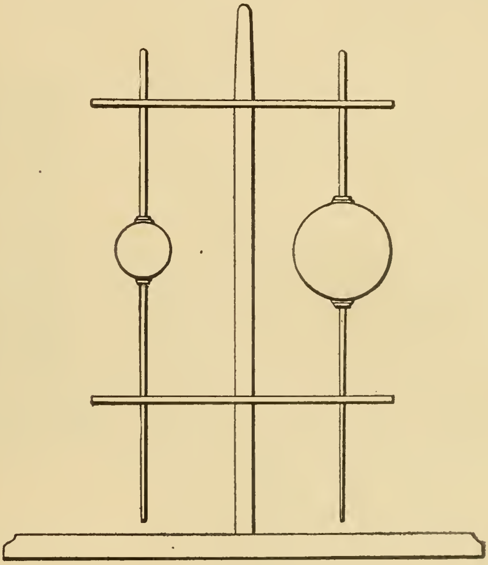


Fig. 40.—The Gillmore Wires.

The Gillmore needles, shown in Figure 40, consist of two wires each supporting a weight, one having a diameter at the bottom of the wire of 1/12-in. and carrying a weight of ¼ of a pound, and the other being 1/24-in. in diameter and carrying a weight of one pound. The cement paste is moulded into a cake or pat, and the time observed when it will sustain these needles without perceptible indentation, the former giving the initial and the latter the hard set. These needles are often furnished without a permanent mounting in which case great care should be exercised to apply them vertically, since applying them at an angle will decrease the area under pressure and hence give false results. For accurate work they should always be mounted in a frame as shown in the figure.

For determinations of hard set, these two forms of appara tus are equally reliable, but, for initial set, the results obtained with the Vicat are generally more accurate, the point when a needle ceases to penetrate a certain depth being much more clearly marked than the point when it ceases to indent the surface. There is a long period during which it is almost impossible to tell whether a surface mark is made or not, since the needle invariably leaves a circular white spot, probably caused by the crushing of some minute crystals, which may readily be mistaken for real penetration. In routine work, either of these forms may be used, with the preference, probably, in favor of the Vicat.* In point of value, the results of both initial and hard set obtained with the Vicat needle will be found to average from a half to three-quarters of the results of the Gillmore wires.†

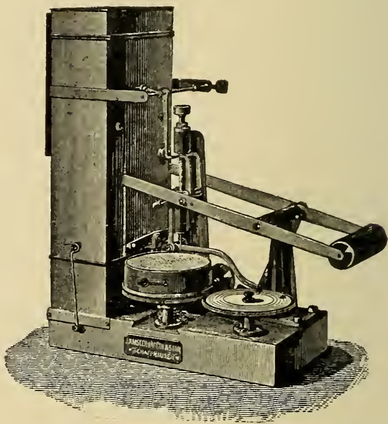


Fig. 41.—The Amsler-Laffon Apparatus for Automatically Determining Time of Setting.

Many contrivances have been made to obtain the time of setting automatically, but none of them has been found capable of use in routine testing. The Amsler-Laffon apparatus (Fig. 41) will automatically record the penetration of a needle and the temperature of the paste at intervals of a minute until the setting is complete. Cornell University has a machine in which a needle is moved over a trough of cement paste, released every minute and the penetration recorded. Such machines, however, are constructed more for experimental purposes, their mechanism being too complicated for every-day use.

Methods of Operation.—In preparing cement pastes for this test it is not only necessary that they should be of a standard consistency, but also that this consistency be obtained by a

*The Vicat needle is recommended by the Committee of the American Society of Civil Engineers. See Appendix A.

†For data on the comparative values obtained from these two forms of apparatus, the reader is referred to the Watertown Arsenal Report, "Tests of Metals, etc., for 1901.

uniform method. The longer a paste is manipulated the wetter and more plastic it becomes, and, furthermore, the properties of cement pastes vary with different lengths of time employed in working, although the consistency be the same. That is to say, that although apparently similar, the properties of a cement paste mixed with a certain amount of water for a short time will be different from one mixed with less water for a longer time. For this reason a uniform method of mixing must be followed, and in practice, it is generally advisable to follow the same method as that used for the making of neat tensile briquettes.*

In making pats or cakes for use with the Gillmore wires, or in filling the moulds for the Vicat needle, care must be taken that the mass of the specimen be uniform throughout. The material must never be tamped, or rammed, since this tends to create a variation in its density, and also to flush the water to the top, which causes errors in surface measurements. The American Society of Civil Engineers' Committee† recommends the following method for filling the moulds of the Vicat needle:

"In making the determination, the same quantity of cement as will be subsequently used for each batch in making the briquettes (but not less than 500 grams) is kneaded into a paste, as described in paragraph 58, and quickly formed into a ball with the hands, completing the operation by tossing it six times from one hand to the other, maintained six inches apart; the ball is then pressed into the rubber ring, through the larger opening, smoothed off, and placed (on its large end) on a glass plate, and the smaller end smoothed off with a trowel."

This Committee also advises that the test pieces be stored in a damp closet‡ during the time of setting, but this is generally found to decrease, rather than increase the uniformity, on account of the frequent removing of the specimens in making the trial tests, and, moreover, has the disadvantage of so much prolonging the time for hard set, that it often exceeds the ordinary working day. In routine testing this proceeding will be unnecessary, provided care is taken to keep the specimens protected from any extreme heat, the sun's rays, or a draught of air that would tend to dry out the water.

---

*See page 117.
†See Appendix A.
‡For description of damp closets, see page 130.

In operating the Vicat needle or plunger for initial set or for normal consistency, the proper method is to bring it carefully into contact with the surface and then quickly release it, and not to let it down gradually into the paste.

The Author's Method.—A sample of 500 grams is weighed and placed on a mixing slab of plate glass, this quantity being sufficient not only for the set test, but also for the making of the test-pieces to be used in the soundness tests described in Chapter X. The cement is formed into a crater and an amount of water about one per cent. short of that ordinarily required to bring that brand of cement to normal consistency, poured into the center, the water being at a temperature of between 65 and 75 degrees Fahr. Material from the edge is turned in with a four-inch trowel until the water is absorbed, and the paste vigorously worked with the hands, as dough is kneaded,* for a minute and a half. Additional water is then slowly added from a burette and mixed in, until normal consistency is reached, and although this process is not strictly accurate it introduces an error so small as to be negligible, and saves the time and trouble of making a separate determination for consistency. Actual determinations on the Vicat plunger for normal consistency are only made at intervals as a check, an experienced operator being able to recognize it and gauge it within a half per cent. at least. The paste is then formed into a ball, forced into the larger end of the rubber ring of the Vicat needle, smoothed on the bottom, placed on a piece of plate glass (4 ins. × 4 ins. × $\frac{1}{8}$-in.), the excess of material cut off the top and smoothed with the trowel without pressing or ramming. The specimen is kept in the open air of the laboratory, the temperature of which is maintained between 65 and 80 degrees Fahr., and readings of the penetration made at intervals of from one to ten minutes as may be necessary. The records cover the percentage of mixing water, the time of adding the water, the time at which initial and hard set are attained, and the temperature of the room at the beginning and end of the test.

It must be understood, however, that this method, while giving sufficiently accurate results with experienced operators, could not be employed by a novice; for any other than an ex-

*The method of kneading is more fully described on page 120.

pert a separate determination of consistency should always be made, and then the proper amount of water added at once for the set test.

Sources of Error and Accuracy.—Excluding the errors due to improper manipulation, which can always be traced to inexperience or lack of proper knowledge, the chief source of error in this test is the subjecting of the sample to improper environment. Many testers collect their samples on one day and test them the following, and during the night leave them near a radiator, or exposed to dampness so that by the following morning their setting properties have entirely changed. Testing in a room too hot or too cold, or using water of an abnormal temperature, is also responsible for many errors, while the use of impure water may produce a chemical action and thus introduce irregularities. In operating the Vicat needle care should be taken to keep it clean and straight, to apply it vertically, near the center of the specimen and not on the edges. The needle must always be brought into contact with the surface of the paste and quickly released; lowering it slowly into the paste will invariably give a shorter period for initial set.

At best, the test of time of setting can be considered as only approximate; a skillful operator should work with a probable error of about 10 per cent., but a novice will often be fortunate to duplicate his results within 30 or 40 per cent.

Interpretation of Results.—On account of the approximate character of the determination, and the necessary presence of the personal equation, the requirements for setting should always be interpreted liberally. A mortar or concrete on actual construction will generally, on account of the wetter mixture, and the presence of the aggregate, require from 2 to 4 times as long to set as the test-piece in the laboratory. Concrete for heavy construction usually requires about 20 minutes to be mixed and placed in the work, and on the hypothesis that the concrete requires twice as long to set as the cement paste, a test of less than 10 minutes initial set would show that the concrete had commenced setting before being tamped into place, and hence had been subjected to reworking, although not retempered. Cement mixed in a mortar box for bricklaying, or similar purposes, will often stand over an hour after mixing and before being used, and if this is allowed, the requirements for setting should be more rigidly adhered to. Generally, if the test of initial set is less than a half or a third of the time required to mix and place the material on the work, the shipment should be rejected, or held for further seasoning.

The determination for hard set is less important, and unless prolonged beyond all reasonable bounds, so that the progress of the work will be delayed, rejection on failure to pass this requirement alone is rarely, if ever, justifiable. Gross failure in this test will almost invariably be accompanied by failure in tensile strength, on which ground it may be rejected without question.

CHAPTER IX.

TENSILE STRENGTH.

The test of tensile strength consists in mixing cement and water, or cement, sand and water into a paste, forming it into test-specimens, called briquettes, which are allowed to set and harden under definite conditions, and then determining the amount of force necessary to cause rupture in tension at the expiration of fixed intervals of time.

The object of the test is to obtain a measure of the strength of the material as used in actual work. In construction, a concrete is often subjected to every conceivable form of stress, except, possibly, that of torsion, while the testing is confined almost exclusively to tension. This condition is the outcome of both theoretical and practical considerations. While it is impossible to formulate definite ratios between the ultimate strengths of cement under different forms of stress, nevertheless, the tensile strength is, more or less, a measure of the compressive, transverse, adhesive and shearing values,* and, furthermore, investigations have apparently shown that the strength of cement in tension is more susceptible to any good or bad influences operating on the material, and hence furnishes a better criterion of its value than tests made in any other manner, the results of the tensile test thus giving the most reliable basis for computing the values of the strength under other forms of stress.

The practical considerations favoring the adoption of this form of strength test are the small and easily handled test-specimens, the lower stress, as compared with compression tests, necessary to cause rupture, and also the fact that uniformity in the preparation of the specimens is only necessary in a small portion of the specimen, namely the breaking section, while accurate test-pieces for the other determinations must be homogeneous and uniform throughout their entire mass.

Although in practice cement is almost invariably mixed with

*For the relations between tensile strength and that of compression, cross-breaking, etc., see Chapter XII.

an aggregate, tests are usually made on both neat cement and sand mixtures. The objection to the use of test pieces of neat cement is that they are not similar to the conditions of practice, while the reason that sand tests are of comparatively recent origin is that the sand introduces another variable in the influence exerted by its character.

The rupture of a neat briquette takes place when the force exerted exceeds the sum of the cohesive strengths of the particles of cement lying in the least section to the adjacent particles. In a sand briquette, on the other hand, rupture is induced by failure in cohesion of adjacent cement particles, by failure in the adhesion of the cement to the sand grains, and by shearing of the cement between overlapping grains of the aggregate. The reason that the strength of briquettes of sand mortar apparently exceeds the sum total of these strengths is that, while a neat briquette ruptures in practically a plane section, a sand mortar fails along an irregular surface due to the projection of the sand grains, so that the actual area over which rupture takes place is very much in excess of the cross-section. Now, since the stresses producing failure in sand briquettes are very complex in character, and since they bear no definite relation to the strength of pure cohesion, and, moreover, since in actual construction cement is commonly used with an aggregate, it follows that the strength of sand briquettes furnishes a better measure of the conditions of practice. Neat briquettes, on the other hand, are more susceptible to both interior and exterior influences, and hence are better criteria of the character of the material. In other words, the sand tests are a measure of strength, while the neat tests are more a measure of quality.

The sand mixture commonly employed for the testing of Portland cements is 1 part, by weight, of cement to 3 parts of sand. The periods at which the briquettes are broken have been arbitrarily fixed by usage at 7 and 28 days, although much longer periods are necessary for the accumulation of reliable data on experimental research. Twenty-four hour tests also are generally made on neat briquettes, and occasionally on sand. Tests after 3 days are frequently made in England and on the Continent, but are rarely employed in this country.

Effect of Composition.—Cement is composed essentially of silicates and aluminates of lime, to which after burning is added a small amount of sulphate of lime. The aluminates and sulphates* are responsible for the setting and early strength, and the silicates for the final hardening. It follows, therefore, that a large proportion of aluminates, and hence usually high sulphates, make cements of greater early strength and, on account of the corresponding decrease in the silicates, lower ultimate strength. Moreover, the strength in the early periods of hardening, due to the aluminates and sulphates, is apparently not permanent in character, but is soon lost, the action being somewhat in the nature of a veneer on the true strength. The diagram (Fig. 42) rather crudely illustrates the idea, it not be-

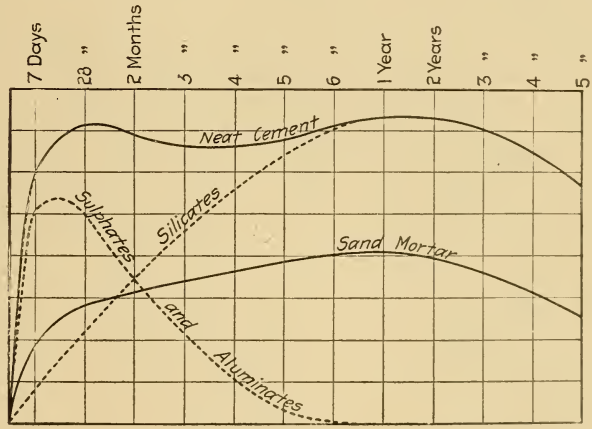


FIG. 42.—Diagram Illustrating the Hardening of Portland Cement, Measured by Tensile Strength.

ing intended to show actual values nor even the relative magnitude of one of the dotted curves to the other, but only that the actual strength is the sum of the strengths of these separate ingredients. The reason for the frequently occurring loss in strength found in periods of from 28 days to 1 year can be thus readily explained. The subsequent retrogression will be discussed later in this chapter. Another important factor in producing high tests at early periods is an excess of lime, and this being the case, it frequently happens that to give good specification tests, a cement is produced so high in lime as to be unsound. For this reason, high tensile tests at 7 days are

*See page 9.

often considered to be indicative of the presence of an abnormal amount of lime, and have occasionally been excluded in specifications which place a maximum as well as a minimum on the seven-day neat tensile test.

Small amounts of magnesia, alkalies, etc., have little or no influence on the strength. Excess of these ingredients, or abnormal amounts of lime and sulphates only affect the strength in affecting its soundness,* since disintegration, either incipient or pronounced must vitally influence the resistance of the material.

Effect of Age.—The effect of the age of cement prior to testing is to lower the early strength and, if prolonged, the ultimate strength of the material. Cement, on standing, gradually absorbs water and carbonic acid from the air, which first attacks the expansives such as free or loosely combined lime, then those ingredients that give the early strength, and lastly, those responsible for the final hardening. A certain amount of storage, usually from a week to a month, is necessary to obtain a sound cement. Further storage gives a cement of lower strength in the early stages of hardening, but affects the values at later periods but slightly, unless too much prolonged. When a cement contains a high proportion of sulphates, the falling

TABLE XXVIII.—Effect of Seasoning on the Tensile Strength of Portland Cement. (Tests by the Author.)

Mixture	Age of Cement Before Test	Tensile Strength, Lbs. per Square Inch: 24 hours	7 days	28 days	2 mos.	4 mos.	6 mos.	12 mos.
Neat Cement	Original	541	739	786	772	732	743	699
	1 month	553	717	769	769	743	762	753
	3 months	504	664	698	732	729	715	721
	6 "	471	622	703	714	694	703	702
	9 "	485	623	673	680	661	701	670
	12 "	382	531	623	655	659	639	672
	18 "	245	502	587	603	602	639	654
1 Cement: 3 Standard Quartz Sand	Original	...	273	329	342	330	355	329
	1 month	...	242	302	339	339	336	341
	3 months	...	247	294	302	307	324	340
	6 "	...	219	273	291	283	307	301
	9 "	...	204	253	293	284	301	294
	12 "	...	167	241	259	243	281	292
	18 "	...	132	187	231	232	245	263

Each value based on 5 briquettes only.

*See Chapter X.

off in early strength is more pronounced.* Material from one to three months old will usually give the best results in practice.

Table XXVIII. gives the results of a series of tests made by the author to demonstrate the effect of age on the strength of cement. The tests were made on a rotary-kiln Portland from the Lehigh Valley district, which was about a month old when the first tests were made, and so was seasoned sufficiently to be entirely sound. The cement was kept in its canvas bag in the ordinary air of the laboratory, and at intervals tested with the results shown.

Effect of Fineness.—The effect of increased fineness is, generally speaking, to increase the strength of sand mortar, and to decrease that of neat cement. It seems natural that the finer a cement is ground the more readily it will be acted upon by water, hence becoming more effective, while the interior of the coarser particles remains practically inert. The finer cement is, therefore, more active, and in a sand mortar will cover the surfaces of the sand grains more thoroughly, and thus will give higher values. The reason for the lower strength of neat briquettes is less apparent, but may be explained by the fact that the coarse particles give a coherence to the mass in furnishing something to which the finer particles can adhere, and that in breaking, rupture takes place around these particles rather than through them, thus increasing the area of the breaking section, as is the case with sand briquettes. Another possible reason may be that in the coarser cement the particles are better graded, and may pack more closely, thus giving a denser mass.

It also follows that, because it is more readily acted upon by water, the finer cement will attain its ultimate or highest strength at an earlier period than the coarse one.

The effect of fineness on tensile strength may be clearly seen in Tables XXIX. and XXX.

The fact that the coarse particles of cement remain practically inert, even after long periods of time, may be proven by regrinding old briquettes and remoulding them, or by fastening together with a rubber band the broken halves of a neat briquette made from a coarse cement, in either of which cases it will be found that considerable strength is developed.

*See page 85.

TABLE XXIX.—Effect of Fineness on Tensile Strength of Portland Cement. (From Butler's "Portland Cement.")

Brand	How Treated	Per Cent. Fineness Residue on:			Time of Setting (minutes)		Percentage of Water for Briquettes		Tensile Strength in Lbs. per Square Inch: Neat Cement					1 Cement : 3 Sand				
		No. 50	No. 76	No. 180	Initial	Hard	Neat	Sand	7 days	28 days	3 mos.	6 mos.	12 mos.	7 days	28 days	3 mos.	6 mos.	12 mos.
A.	As received from manufacturer..	4.0	16.0	33.0	15	90	21.66	7.81	483	572	623	662	653	183	276	383	440	482
	Reground extremely fine	0.0	0.0	2.5	4	60	25.00	9.38	408	541	538	531	506	347	452	564	509	637
	All particles not passing 180 sieve removed, and sand of similar size substituted....................	..	..	..	..	..	18.33	7.81	418	456	567	596	650	153	210	272	337	386
B.	As received from manufacturer..	8.0	20.0	35.0	10	90	20.00	7.81	405	618	622	694	759	187	245	334	377	392
	Reground extremely fine	0.0	0.0	1.0	1	4	25.00	8.13	540	474	560	466	477	282	363	494	595	617
	All particles not passing 180 sieve removed, and sand of similar size substituted....................	..	..	..	..	..	18.33	7.81	403	448	602	678	714	158	209	303	348	378
C.	As received from manufacturer..	4.0	11.0	28.0	8	60	19.16	7.81	445	493	584	663	706	167	230	312	373	399
	Reground extremely fine	0.0	0.0	0.8	2	5	26.66	8.13	433	501	514	482	535	287	364	508	585	599
	All particles not passing 180 sieve removed, and sand of similar size substituted....................	..	..	..	..	..	18.33	7.81	367	453	604	669	692	145	212	271	348	405
D.	As received from manufacturer..	2.5	15.0	39.0	20	120	20.00	8.59	592	630	736	791	751	240	297	389	425	410
	Reground extremely fine	0.0	0.0	0.8	2	10	30.17	11.26	417	394	459	476	498	387	465	560	585	618
	All particles not passing 180 sieve removed, and sand of similar size substituted....................	..	..	..	..	..	18.33	7.42	470	565	653	698	754	200	246	312	382	380

Effect of Environment.—The environment of the cement prior to testing exerts less influence on the strength than on the time of setting. High or low temperatures have little effect, provided the sample is brought to normal when the test is made. Excessive dampness is equivalent to a longer period of seasoning, provided it is not sufficient to cause actual hydration.

The temperature at which the briquettes are made exerts a somewhat greater influence, but for the ordinary ranges occurring in the laboratory the effect is practically negligible. In regard to lower temperatures, Sabin* says: "It appears that the briquettes made in a low temperature (34° to 37° Fahr.) are usually stronger than those made in the ordinary temperature of 65° to 68° Fahr." The table of tests accompanying this statement, however, shows the differences to be but slight and not altogether consistent. It is advisable, nevertheless, to keep the temperature of the laboratory as near 70° Fahr. as practicable.

TABLE XXX.—The Effect of Fineness on Tensile Strength of Portland Cement.
(A Short Series of Tests by the Author.)

No.	How Treated	Per Cent. of Water	Tensile Strength 7 days	Tensile Strength 28 days
1.	Neat cement	21.0	540	591
2.	Neat briquettes of material passing No. 200 sieve	22.0	481	502
3.	1—cement : 1 part standard quartz sand	12.1	479	653
4.	1—No. 2 material : 1 " " " "	12.4	456	683
5.	1—cement : 2 parts " " "	10.3	338	507
6.	1—No. 2 material : 2 " " " "	10.6	423	532
7.	1—cement : 3 " " " "	9.4	169	243
8.	1—No. 2 material : 3 " " " "	9.7	283	357
9.	1—cement : 3 " bar sand	9.6	120	163
10.	1—No. 2 material : 3 " " "	9.8	181	223
11.	Cement in which the material retained on the No. 200 sieve was replaced by sand of same size	17.6	493	557
12.	1—part No. 11 material : 3 parts standard quartz sand	9.0	164	235
13.	Cement passing No. 100 and retained on No. 200 sieve	20.0	130	253
14.	Cement passing No. 50 and retained on No. 100 sieve	16.0	..	24

Fineness of neat cement:—No. 50—0.0%; No. 100—9.2%; No. 200—24.8%.
Each value average of 5 briquettes.

*In "Cement and Concrete," by L. C. Sabin.

THE FORMING OF BRIQUETTES.

Amount of Mixing Water.—Tables XXXI. and XXXII. show the effect of different percentages of mixing water on the tensile strength of neat and sand briquettes. For the ranges that are practicable for purposes of testing, it will be found that for early periods, dry briquettes give the higher values, but that ultimately the wetter mixtures generally equal and occasionally even exceed them. As stated in Chapter VIII., it has been shown that different cement mixtures have more nearly similar properties when mixed to the same degree of plasticity than when mixed with a fixed amount of water. The amount of

TABLE XXXI.—Effect of Varying Percentages of Water on the Strength of Portland Cement. (Tests from paper on the "Tensile Strength of Cement," by E. S. Larned, Proc. Am. Soc. Test. Mats., 1903.)

Brand	Water Per Cent.	Sieve Test: Residue on No. 50	No. 100	No. 180	Wire: Minutes Light	Heavy	Tensile Strength 24 hours	7 days	28 days	3 mos.	6 mos.	12 mos.
A......	13	0.1	7.0	18.0	13	270	366	775	859	1067	892	832
	14				18	303	404	780	891	972	852	781
	16				22	327	363	602	725	844	806	723
	18				15	383	308	570	723	785	728	724
	20				56	703	225	590	718	760	674	636
	22				52	833	166	554	649	731	643	604
	24				188	918	42	510	691	695	632	574
B......	15	0.15	5.4	21.2	12	207	371	655	875	941	720	787
	16				29	297	303	750	973	1008	735	816
	18				80	355	260	649	773	831	645	748
	20				142	402	233	500	693	716	621	676
	22				268	473	184	546	635	658	601	589
	24				327	912	167	539	649	644	629	755

NOTE.—Each Value is Average of Six Briquettes.

water required to bring different cements to the same consistency varies with the composition, age, fineness, etc., so that the proper amount must be determined experimentally in each case. Methods for determining normal consistency have already been discussed,* and either a consistency obtained by the ball method, or that recommended by the Committee of the American Society of Civil Engineers† will give good results, although the former will be found somewhat easier to manipulate. Whatever consistency be adopted, it is advisable to use the same for both time of setting and neat briquettes. The use of a

*See page 93.
†See Appendix A.

fixed percentage of water for all cements, such as is given in the specifications of the U. S. Army Engineers* is incorrect in theory and difficult in practice. The average Portland cement requires about 20% of water to bring it to the consistency required by the ball method, and about 21% for the Am. Soc. C. E. Committee method.

One great advantage in the use of wetter mixtures is that the moulds can be filled more uniformly by hand, since it is almost impossible to compact very dry briquettes similarly; but, on the other hand, the wet briquettes are difficult to manipulate properly, are liable to shrink in the moulds, and often contain large

TABLE XXXII.—Effect of Variations in the Consistency of Mortar on the Strength of Portland Cement.
(From Sabin's "Cement and Concrete.")

Parts Sand to 1 Cement by Weight	Tensile Strength, Pounds per Square Inch, for Consistency Number								
	1	2	3	4	5	6	7	8	9
0	608	635	763	744	708	707	729	685	...
1	513	543	618	588	594	613	566	566	538
2	...	...	429	...	447	398	393	382	...
3	...	289	...	322	329	310	...	279	...
5	...	208	...	230	201	189	...	167	...

All Tests made at 3 Months. Sand—Crushed Quartz

Consistency: Significance of Numbers: Increasing Per Cent. Water Used for Higher Numbers:
- 1.—Very Dry; Little or no Moisture Appeared on Surface of Briquettes.
- 5.—About Proper Consistency for Briquettes.
- 9.—Very Moist; Mortar would barely Hold Shape, and Shrank in Moulds in Hardening.

air bubbles which are responsible for low results. The normal consistency recommended gives neither the highest nor the most uniform values, but is an excellent mean between the two, and at the same time is most convenient for manipulation.

In mixtures of cement and sand, the amount of water required to produce normal consistency is much more difficult to obtain. It is practically impossible to determine its consistency by direct measurement, the mixture being too incoherent for the use of the ball method, and the sand grains not permitting of determinations by penetration. Since the practice of using a fixed percentage of water for any mixture is just as incorrect for sand mortars as for neat pastes, although the effect is not so great, recourse must therefore be made to a formula by which

*See Appendix D.

the proper amount of water may be found, if the neat consistency has been determined.

Several of these formulas have been proposed, although no one of them can be recognized as a standard. Feret's formulas are among the best known, and were evolved empirically from the plotting of curves representing the average judgment of several operators. These formulas are:

For mortars of plastic consistency:

$$E = \frac{2}{3} NA + 60$$

and for mortars of dry consistency:

$$E = \frac{2}{3} NA + 45$$

in which E = weight of water in grams required for one kilogram of dry mixture of cement and sand;

N = weight of water in grams required for one kilogram of neat cement; and

A = weight in kilograms of cement in one kilogram of the dry mixture.

The first formula gives the consistency generally used for hand mixing, while the second gives a consistency suitable for mechanical apparatus such as the Böehme hammer. While the first formula is well adapted for the average range of practice, it will be found that the extreme values are in considerable error. Moreover, it is impossible to vary the formula consistently for the use of different sands.

The author has attempted to evolve a formula for the consistency of sand mortars from purely theoretical considerations but was unsuccessful, the great difficulty being due to the varying void spaces in the different mixtures. However, by slightly altering the form and by introducing empirical constants, a formula was evolved which has given entire satisfaction in practice for over three years.

The formula is

$$x = \frac{3\ N + Sn + 1}{4\ (n + 1)}$$

in which

x = per cent. of water for sand mixture;

N = predetermined percentage of water required to bring neat cement to normal consistency;
n = parts of sand to one of cement by weight;
S = a constant depending on character of sand and consistency desired.

The empiric constants used in this formula were obtained from the results of almost 2,000 tests covering the greater part of one winter's experimental work.

This formula has the advantages of being applicable to any mixture from 1 : 1 to 1 : 5, is adaptable to any sand, and may be altered to give any desired consistency. For ordinary processes of hand moulding, the constant S becomes 30 for standard quartz sand, making the formula read:

$$\frac{3\,(N + 10\,n) + 1}{4\,(n + 1)}$$

the values for which are given in Table XXXIII.

For Ottawa sand, S = 25, while for the bar and bank sands ordinarily used in construction, S varies from about 27 to 33.

TABLE XXXIII.—Percentages of Water to Use in Mixtures of Cement and Standard Quartz Sand—Based on Formula, $\frac{3\,(N + 10\,n) + 1}{4\,(n + 1)}$

Neat	1.1	1·2	1·3	1:4	1:5	Neat	1:1	1:2	1:3	1:4	1:5
15	9.5	8.8	8.5	8.3	8.2	26	13.6	11.6	10.6	10.0	9.5
16	9.9	9.1	8 7	8.5	8.3	27	14.0	11.8	10.8	10.1	9.7
17	10.3	9.3	8.9	8.6	8.4	28	14.4	12.1	10.9	10.3	9.8
18	10.6	9.6	9.1	8.8	8.5	29	14.8	12.3	11.1	10.4	9.9
19	11.0	9.8	9.3	8.9	8.7	30	15.1	12.6	11.3	10.6	10.0
20	11.4	10.1	9.4	9.1	8.8	31	15.5	12.8	11.5	10.7	10.2
21	11 8	10.3	9.6	9.2	8.9	32	15.9	13.1	11.7	10.9	10.3
22	12.1	10.6	9.8	9.4	9.0	33	16.3	13.3	11.9	11.0	10.4
23	12.5	10.8	10.0	9.5	9.2	34	16.6	13.6	12.1	11.2	10.5
24	12.9	11.1	10.2	9.7	9.3	35	17.0	13.8	12.3	11.3	10.7
25	13.3	11.3	10.4	9.8	9.4	36	17.4	14.1	12.4	11.5	10.8

If it is desired to use a somewhat drier consistency adaptable for use with mechanical moulders, S may be reduced to 26 or 27 for standard quartz, and to 21 or 22 for Ottawa sand, and it will be found that the consistencies obtained are practically uniform throughout the entire range as given in the table. In routine testing with standard quartz sand the values given in Table XXXIII. will be found convenient and satisfactory.

Temperature of Mixing Water.—For the ordinary ranges liable to occur in a laboratory, the temperature of the mixing water has little if any effect upon the strength. Extremely cold water, however, will retard the process of hardening, and hot water accelerate it, but it requires a very decided variation to affect the results appreciably. It is advisable, nevertheless, to insure against error by always using water as near to 70 degrees Fahr. as practicable.

Purity of Mixing Water.—Small amounts of salts in solution or impurities in suspension generally have but little effect on the strength. Care, however, should be taken that the water is neither acid nor strongly alkaline. Water from the storage tanks is often very alkaline, and, if used in mixing, may have an appreciable influence on the results. Sea-water should never be used in routine tests, although M. Alexandre* states that there is little difference in the strength of mortars gaged with fresh and with salt water. Suspended mineral or organic matter in sufficient quantity may introduce errors of no small magnitude, especially in tests of sand mortar.

Sand.—It is scarcely within the province of this book to enter in detail into the effect of different varieties of sand on the strength of the resulting mortar.† It will be sufficient to state that ordinarily a rather coarse sand will give higher results than a finer one, and that a sand whose grains are graded in size will surpass either one, and furthermore that the differences in strength caused by the use of different grades of sand may amount to as much as 200 or 300 pounds. For purposes of routine laboratory testing therefore, it is necessary to employ a standard sand, if the results obtained in different laboratories are to be at all comparable.

The sand commonly employed in the United States for cement testing is artificially prepared crushed quartz, sifted to pass a sieve of 20 meshes to the lineal inch and to be retained on one of 30 meshes. The use of this sand was proposed in 1885 by the Committee of the American Society of Civil Engineers. As sold, the sand is never sifted clean, and therefore must either be

*Annales des Ponts et Chaussees, 1890.

†For comprehensive data on the effect of the granulometric composition of sands on the strength of mortar, the reader is referred to "Cement and Concrete," by L. C. Sabin; "Concrete—Plain and Reinforced," by F. W. Taylor and S. E. Thompson; "Cements, Mortars and Concretes," by M. S. Falk, and "Materials of Construction," by J. B. Johnson.

sifted again in the laboratory, or else the sand as sold must be required to pass a limiting specification. It has been the author's practice to specify that not more than 3 per cent. of this sand shall either pass the No. 30 sieve or be retained on the No. 20, and then, if fulfilling these requirements, to use the sand as received without further sifting.

Although, if sufficient care be exercised, this sand may be procured of very uniform character, there are nevertheless several serious objections to it—the angular character of the grains which make it difficult to compact the mortar closely, the high percentage of voids (40 to 45 per cent.), and the expense and occasional difficulty of procuring it.

For these reasons, the later Committee of the American Society of Civil Engineers has recommended the use of a natural sand from Ottawa, Illinois, sifted, as with the crushed quartz, to 20-30 size. This is a pure silicious sand, having grains almost spherical in shape, a void space of 30 to 35 per cent., and may easily be compacted into a dense mortar. Briquettes, broken at 7 and 28 days, made of one part cement to three parts of this sand will average from 20 to 30 per cent. higher than those made similarly from crushed quartz. If this sand is employed therefore, the specification requirements for mortar briquettes should be increased by 20 or 25 per cent. Although Ottawa sand has several advantages over crushed quartz, its use at the time of this writing is by no means general, chiefly for the reason that the majority of the present data on cements, and also the greater number of existing specifications, are based upon results obtained with crushed quartz.

For field laboratories situated in places where it is difficult and expensive to procure either of these sands, it is permissible to employ a local sand, which has first been carefully tested in comparison with the standard sand on which the specifications are based. The sand should always be sifted to a definite size, preferably 20-30, since every natural sand varies considerably in different parts of the bed.

The sieves for sand testing should be carefully calibrated in a manner similar to those used for testing the fineness of cement,* and the wire should be of the following sizes: No. 20, 0.0165 inches; No. 30, 0.0112 inches.

*See page 68.

The standard sands of England, France and Germany are natural sands occurring in definite localities, and sifted to a size nearly equivalent to 20-30. In France a compound or graded sand is also employed in certain tests. German normal sand gives results averaging 5 or 10 per cent. stronger than crushed quartz.

Form of Briquette.—The standard American form of tensile briquette is shown in Fig. 43. This form was adopted by the Committee of the American Society of Civil Engineers in 1885, and endorsed by the later Committee except for the rounding off of the corners, which makes it more easy to fill, handle, and

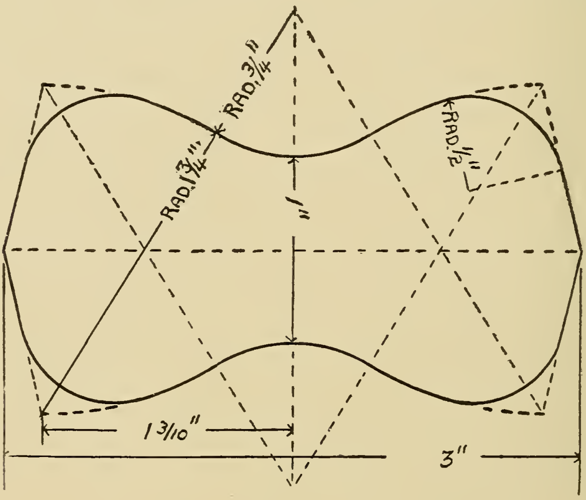


Fig. 43.—American Standard Form of Briquette.

remove from the moulds. The standard English briquette* is practically identical. Each has a cross section of one square inch. The objections to this form of briquette are first, that the angle between the bearing surfaces is too small, thus inducing lateral compressive and cross-breaking strains; secondly, that the reduction in area at the least section is insufficient, and also that the distance between the least section and the plane of the bearings is not great enough to ensure an equal distribution of stress† in the former.

*See Fig. 140, Appendix E.
†For a mathematical discussion of the distribution of stress over the least section of a briquette, see Johnson's "Materials of Construction," Chap. XXI.

The German standard briquette (Fig. 44), also adopted as the French standard, having an area of 5 square centimeters at the breaking section, is superior to the American form in the angle of the bearing surfaces, but, on the other hand, has too sharp a reduction of area at the least section, which, while it insures all breaks occurring in that place, makes the distribution of stress very unequal and hence gives lower values. The more clumsy shape is another factor in its disfavor. Comparative tests show that the American form of briquette gives results 10 to 20 per cent. higher than the German.

While the defects of both of these briquettes are well recog-

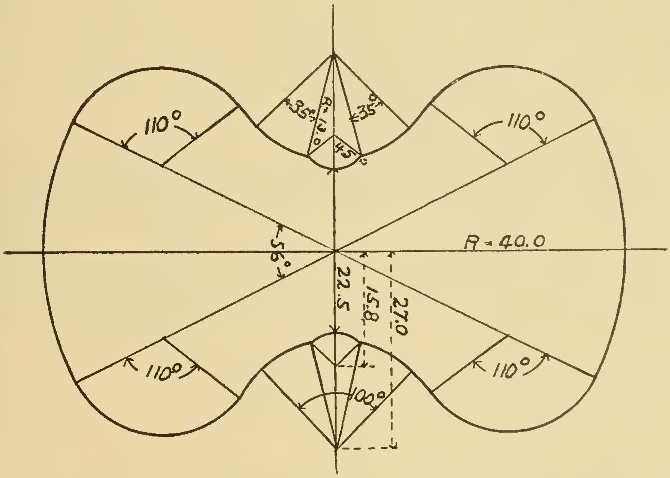


Fig. 44.—The Standard Form of Cement Briquette Used on the Continent of Europe.

nized, it is doubtful whether they will be altered for some time to come, on account of the expense, difficulty and confusion necessarily following such a change.

Moulds.—Moulds for tensile briquettes are made almost universally of brass. Cast iron is occasionally used, but moulds of this material soon rust and become unfit for use.

Moulds are made either single or in gangs of three, four, five, and even ten, but when over four or five are, however, unwieldy and expensive to handle and maintain. Gang moulds of three and four will be found to give the best results in practical work, and are the most economical. Single moulds are filled comparatively slowly, even under the most expert manipulation, while larger gangs must either be very heavily made or else will soon spread in the center, thus destroying the accuracy of the cross-section. Large gangs are usually provided with one or more clamps or bolts in the middle, but these require time to manipulate and make the mould awkward to handle. A plain mould $3\frac{1}{4}$ ins. wide in gangs of three or four will maintain its shape without spreading for several years. To give additional rigidity, the sides of the moulds are sometimes channel shaped, and although wider and hence stiffer are somewhat lighter. The additional width, however, makes handling the mould more awkward, and, for routine work, it is believed that little is gained by their use.

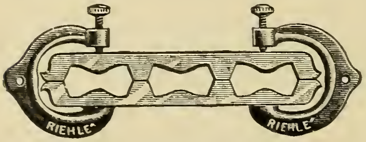


FIG. 45.—Briquette Moulds.

The clamps holding the two parts of the mould together are of

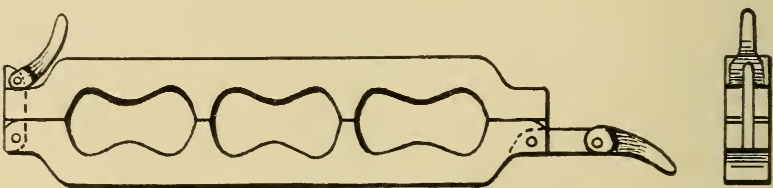


FIG. 46.—Type of Briquette Mould Recommended by the A. S. C. E. Committee

several forms. The screw clamp shown in Fig. 45 admits of greater rigidity, but is very clumsy in manipulation and requires unnecessary space in the damp closet. The end clamp used in the form of mould recommended by the Committee of the American Society of Civil Engineers (Fig. 46) is simple and very convenient. Fig. 47 shows another form of end clamp which is neither as simple nor as easily operated as that of Fig. 46. When end clamps are used, they should always be fastened to the same half of the mould as in Fig. 46 and never opposite each other like those of Fig. 47. The two halves of the moulds are either hinged together at one end, or made separately and fitted together over dowel pins. Either type may be used for single moulds, but the halves should always be separate in gangs.

After much experimenting the author has adopted the use of the form of mould shown in Fig. 46 made in gangs of three and four. In filling and turning over, there are no clamps to get in the way, and in placing them in the damp closet, they can be packed very closely together and can be stood on their sides. In removing the briquettes, the moulds are placed side by side and the clamps all loosened together by two or three blows with a flat iron bar.

They are cleaned by first scraping the sides with a six inch pointing trowel; the halves are then separated and placed side by side along a table, the briquette holes forming long grooves along the line. The inner surfaces are then cleaned all at once by first scraping them with an iron bristle brush of the kind called a "sink scrub brush," and then by rubbing with a piece of oily waste. Every other half is then placed on its mate, and the clamps fastened by knocking them down with the iron bar

FIG. 47.—Another Form of Briquette Mould.

previously described. The moulds used for making 200 briquettes may by this method be cleaned by one man in about half an hour. The life of a 3 or 4 gang mould used daily in this manner is about 2 or 3 years.

Methods of Mixing and Moulding.—In the uniform mixing and moulding of cement briquettes is encountered the greatest difficulty in the testing of cement, primarily on account of the great influence of the factor of personal equation. It is impossible to describe on paper a method by which absolutely uniform results may be obtained unless such elaborate apparatus is employed that the method becomes impracticable for the ordinary laboratory. At best, therefore, any hand method produces results that are comparative among themselves rather than absolute.

The first attempt in this country to prescribe a uniform method for making briquettes was made by the Committee of the American Society of Civil Engineers in 1885, and was as follows:

"The proportions of cement, sand and water should be carefully determined by weight, the sand and cement mixed dry, and the water added all at once. The mixing must be rapid and thorough, and the mortar, which should be stiff and plastic, should be firmly pressed into the moulds with a trowel, without ramming, and struck off level."

On account of the many obvious ambiguities in these rules, the later Committee in 1903 recommended the following method,* which is practically an elaboration of the preceding:

"The material is weighed and placed on the mixing table, and a crater formed in the center, into which the proper percentage† of clean water is poured; the material on the outer edge is turned into the crater by the aid of a trowel. As soon as the water has been absorbed, which should not require more than one minute, the operation is completed by vigorously kneading with the hands for an additional 1½ minutes, the process being similar to that used in kneading dough. A sand glass affords a convenient guide for the time of kneading. During the operation of mixing. the hands should be protected by gloves, preferably of rubber."

"The moulds should be filled at once, the material pressed in firmly with the fingers and smoothed off with a trowel, without ramming; the material should be heaped up on the upper surface of the mould, and in smoothing off, the trowel should be drawn over the mould in such a manner as to exert a moderate pressure on the excess material. The mould should be turned over and the operation repeated."

A method employed by several laboratories for mixing is, after the water has been added and absorbed and the materials formed into a pile, to take a large trowel and starting from the edge to work through the pile, scraping it down little by little with the edge of the trowel under slight pressure. This, however, requires the expenditure of considerable time (to reach the same degree of plasticity about twice as long as the kneading method), and experiments by the author have shown it to be productive of less uniform and accurate results. For very quick setting cements the method is almost impossible.

*See Appendix A.
†See page 93.

Mr. Sabin* recommends for mixing the use of an iron box with a sloping bottom, in which the mortar is worked with a hoe. "The box is 2 feet 7½ inches long, 6 inches wide at the bottom, and at the center is 6 inches deep. The level part of the bottom is 3 inches by 6 inches, and from this level part the inclined portions of the bottom slope up toward the ends at an inclination of about 22½ degrees. The sides of the box extend below these inclined planes to give a level bearing for the box when in use. It is also well to have the sides flare enough to give a width of 6½ inches at the top to prevent the hoe from becoming wedged. A 'German clod hoe,' which is strong and heavy, yet a trifle flexible in the blade, is used in connection with the box."

"The weighed quantities of the dry ingredients being put in the box and well mixed, the measured volume of water is added. Two minutes of hard work, in which the operator may put all his strength, is sufficient to bring the mass to plasticity if the amount of water added is correct."

The author has experimented with this box at some length and found it crude, tedious, awkward, and no better than hand methods in regard to the elimination of personal equation, although Mr. Sabin says "A return to the trowel and slab method of mixing is not likely after a trial of this simple device." It is improbable, however, that a device of this sort would ever take the place of the simple and effective hand methods generally in vogue.

The standard methods of the United States Army,† following German practice, recommend filling the moulds by tamping with a hammer. It is not believed, however, that tamping methods, unless mechanical, secure any greater uniformity of results than compacting under hand pressure, and the objection of the time required for this process is a serious one. Filling briquette moulds under blows from a hammer is a very uncommon practice in this country.

Many other methods of hand mixing and moulding are employed here and there, but none have any recognized status. The trend seems to be more and more to adapt the hand kneading process, and in following the only recognized standard

*In "Cement and Concrete," by L. C. Sabin, p. 106.
†Professional papers No. 28, Corps of Engineers, U. S. Army.

method of the United States, to arrive at greater uniformity both in methods and results.

The Author's Method.—The method outlined by the recent Committee of the American Society of Civil Engineers has been used by the author for several years except that a slightly drier consistency (determined by the "ball" method) is taken, and the mixing is only continued for one, instead of 1½ minutes, and after repeatedly experimenting with many other methods and variations, this method has been found to be the most efficient in routine work. It is not claimed that different operators in different laboratories can obtain even substantially similar results, but a single operator, or several working together can, when

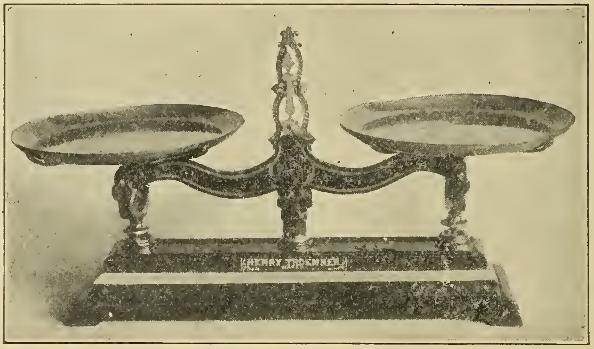


FIG. 48.—Scales for Cement.

employing this method, soon duplicate their results with considerable accuracy, and can day after day make uniform and accurate tests. The speed of making briquettes in this manner is probably greater than in any other, which is a most important consideration in routine work.

On a scale (Fig. 48), sensible to ½ a gram, are first weighed 1,000 grams of the ingredients, this quantity being just sufficient to make eight briquettes, and a convenient amount to manipulate. The cement is formed into a crater and the predetermined quantity of water poured into the center. The author employs the normal consistency as obtained by the ball method,* after one minute of kneading. It is essential that the kneading be always continued for a definite time, since the plas-

*See page 93.

ticity of the paste increases with the time of working, and it is believed that one minute of hard working is amply sufficient to obtain a uniform mixture.

When sand mortars are gauged, the cement and sand are first thoroughly mixed dry (Fig. 49), by hand and trowel, until the pile is of a uniform color, then formed into a crater, and the amount of water given in Table XXXIII.,† poured into the center (Fig. 50). Material from the edge of the crater is turned into the center until the water is all absorbed, then the mixture is turned over loosely with the trowel two or three times to dis-

FIG. 49.—Making Briquettes—Mixing the Dry Ingredients.

tribute the wetted portions evenly, and finally formed into a rounded pile ready for kneading. A six-inch "pointing trowel" is the best form for mixing.

The proper kneading of the mixture is extremely difficult to describe and yet is essential for correct manipulation. 1,000 grams of material, after the water is added and absorbed, form a pile which can just be comfortably covered with the two hands. The kneading is performed by placing the fingers across the pile and pushing the base of the hand towards them while exerting a downward pressure (Fig. 51). A fair idea of the motion may be obtained if the reader will place his hands

†See page 111.

on a table, arched so that only the ends of the fingers and the base of the hands by the wrists are touching, the thumbs off the table and crossed on each other above the back of the hands and the forefingers almost in contact, and then, without moving the fingers, push the wrists quickly towards them, pressing

FIG. 50.—Making Briquettes—Adding the Water.

FIG. 51.—Making Briquettes—Kneading.

down at the same time. The movement of the wrists is repeated five or six times without changing the position of the fingers; then the pile, which is now spread in a line across the direction of working is rounded, turned through 90°, and the kneading repeated. The pile should be worked, rounded and turned about sixteen times in a minute. The downward pressure exerted should be about 10 or 15 pounds.

In filling the moulds enough material to about half fill them

FIG. 52.—Making Briquettes—Filling the Mould.

FIG. 53.—Making Briquettes—Compacting the Mortar.

is first introduced and distributed evenly over the bottom with the fingers and thumbs (Fig. 52), but without exerting any appreciable pressure; this will be found necessary to make the mass of the briquette homogeneous. An excess of material is then placed in and on the mould, extending about half an inch above it, and pressed in firmly with the thumbs, without ramming. In filling a gang mould it is turned to point away from the operator, and then starting from the far end pressed with both thumbs (Fig. 53), 3 times in each briquette, once in each head and once in middle. The pressure exerted should be about 25 to 30 pounds. The mould is then turned back through 90°, an excess of material again placed on top, over which a trowel is drawn several times under a pressure of about 5 pounds, each time cutting off more and more of the excess material until it is flush with the surface of the mould (Fig. 54).

FIG. 54.—Making Briquettes—Troweling the Surface.

The material remaining on the sides of the top is then scraped off with the edge of the trowel, and the briquettes smoothed with two or three more strokes.

The mould is then lifted from the table with a sliding motion, turned over, an excess of material placed on the original bottom, now the top, and surfaced with the trowel as before. The moulds containing neat briquettes, after being surfaced on the first side, and lifted from the table, are placed on strips of glass 4 inches wide, $\frac{3}{4}$-inch thick, and of a length suited to the racks in the damp closet,* and are surfaced for the second time on these strips of glass, which are then placed in the damp closet. The moulds containing briquettes of sand mortar are

*See page 131.

surfaced both times on the mixing table and then placed in the closet on their sides. It is advisable to leave the briquettes in the moulds during the entire time they are in the damp closet, but, if necessary, they may be removed as soon as thoroughly hardened.

The mixing of cement pastes and mortars should always be performed upon a slab of glass, slate or other non-absorbing surface; glass will generally be found the most satisfactory on account of the ease of keeping it clean. A convenient mixing

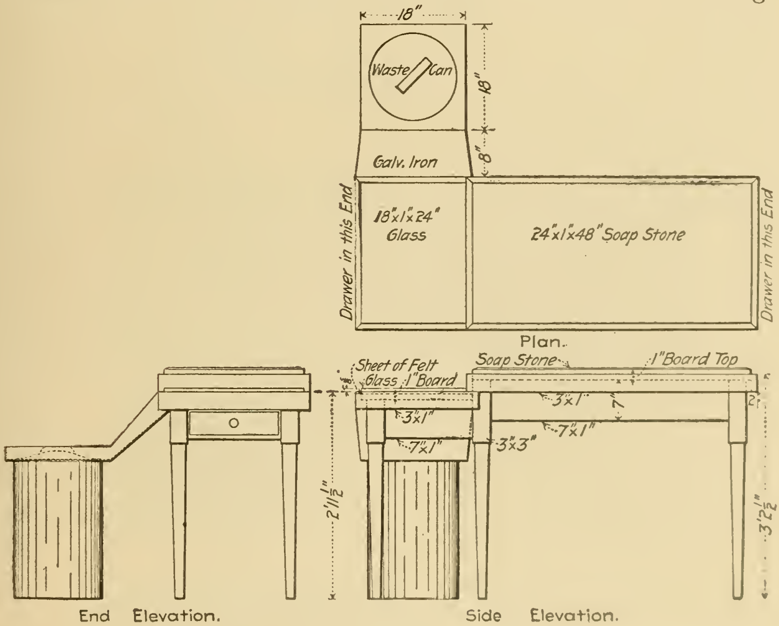


FIG. 55.—Sketch of Mixing Table Used in the Philadelphia Laboratories.

table for laboratory use is shown in Fig. 55, the mixing slab being of glass, and the top of the larger part of soapstone. A mixing slab may be placed on each end, if the volume of work requires the constant services of two operators. For small laboratories a plate of glass, two feet square and ¾-in. thick, fastened on an ordinary table is sufficient.

The hands should always be protected by rubber gloves, when mixing cement, or the lime contained in it will, after a time, make them extremely sore; and the paste also will get under the finger nails, from which it is often very difficult to remove.

Mechanical Mixing and Moulding.—Many devices for the mechanical mixing and moulding of briquettes have been proposed, among the simplest of which may be mentioned ice cream freezers, milk-shake apparatus, and sausage choppers, all of which have been tried for mixers, while many forms of presses and tamping devices have been employed in forming briquettes.

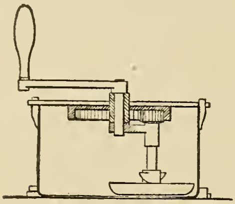


Fig. 56.—Faija's Mortar Mixing Machine.

One of the first of the mixing machines (Fig. 56) was designed by Henry Faija and consists of a pair of paddles revolving in the mixing pan. The author has modified this machine (Fig. 57), adapting it to practical use in enclosing the gearing, thus pre-

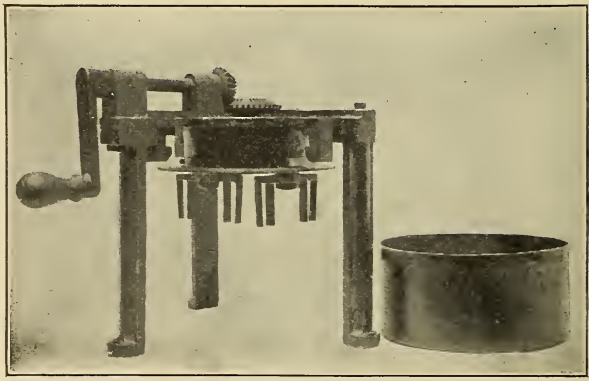


Fig. 57.—Improved Form of Mortar Mixer Designed by the Author.

venting it from becoming clogged, and in making the mixing pan removable, thus much simplifying the cleaning of the pan and paddles, which are brushed down with a bristle brush. The materials are placed in the pan, attached to the machine, turned 20 times dry, then water inserted through a funnel and turned 40 times. The mixing is very complete, but there is little of the working of the mortar necessary to the production of good results.

Steinbruch's mortar machine (Fig. 58) is effective in working the mortar, which is accomplished by means of a wheel revolving in a groove in the pan under which the material is forced by means of blades. A disadvantage of this machine, however, is that the materials must first be hand mixed before being introduced. A combination of this machine with the preceding would give excellent and uniform results, if time could be afforded for so elaborate a process.

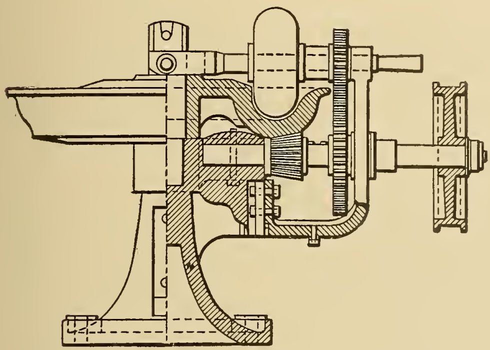


FIG. 58.—Steinbruch's Machine for Working Cement Mortars.

Of the briquette forming machines, that shown in Fig. 59, an American adaptation of the German Böehme hammer, is one of the best known. In this device, a hammer of fixed weight is made to fall on a disc placed over the mould containing the briquette. The number of strokes per minute is fixed, and the machine is automatically stopped at the end of a definite number of blows. The machine is also made in batteries of two, three or four hammers for the simultaneous treatment of several briquettes.

Another simple device on the lines of a stamp mill and on the principle of the hammer form is shown in Fig. 60. The material

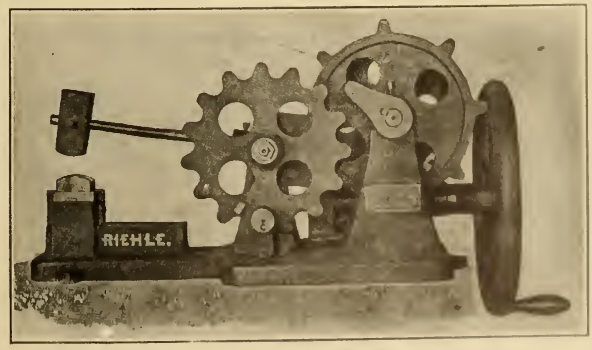


FIG. 59.—Briquette Machine of the Hammer Type.

is placed in a double mould, the upper being used as a guide and to hold the excess material, and given a fixed number of blows.

Machines for compacting briquettes under static load are also employed. That shown in Fig. 61, by turning the crank, applies a static load of either 250, 500, 750 or 1,000 pounds. When the desired load is reached, a clutch is automatically released and a second engaged which releases the load, although the crank is turned in but one direction throughout the entire operation. Another machine of 4,000 pounds capacity is illustrated in Fig. 62, in which the load is applied and released by the hand wheel, and its amount indicated on the spring dial.

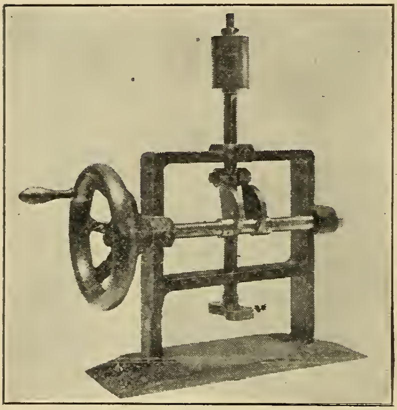


Fig. 60.—Briquette Machine of the Stamp Mill Type.

The author has experimented at some length on these different mixing and moulding machines, and found that none of them is adapted to the requirements of routine testing. They require much more time to operate, and at best give results that are but a very slight improvement in uniformity over ordinary hand methods. Briquette formers operating under static load are extremely unsatisfactory, it being impossible to prepare a briquette of uniform density even under a load of 4,000 pounds unless made very wet, in which case great difficulty is experienced in removing them from the machine. Hammer formers give better results, but are even slower to operate and are entirely impracticable where many tests are to be made unless several extra men are employed for operating these machines alone. Moreover, the author's tests on the best of these machines gave a probable error in the results of 3% against only 4% for hand

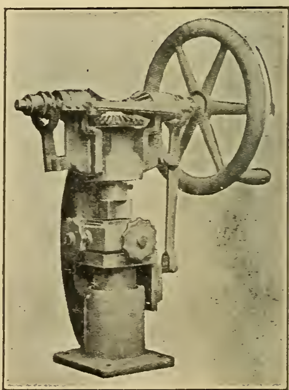


Fig. 61.—Machine for Forming Briquettes Under Static Load.

methods, so that the increased accuracy is disproportionate to the time expended. In routine testing, therefore, these machines have no place. In experimental work, however, they may

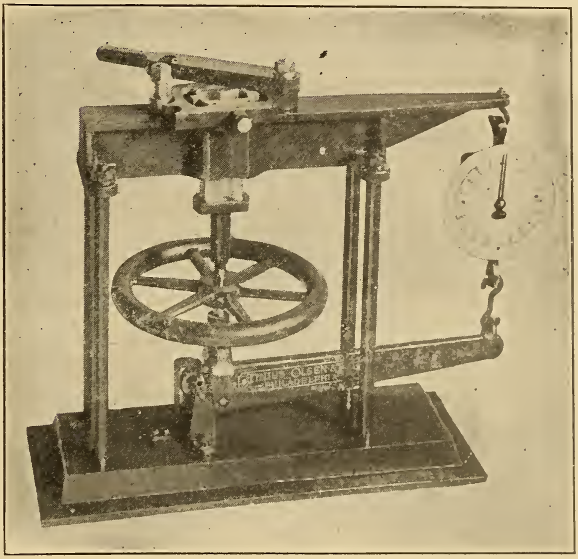


FIG. 62.—Machine for Forming Briquettes Under Static Load.

be employed to advantage, a combination of the modified Faija mixer, the Steinbruch worker, and the hammer former giving the most uniform results. Figure 63 shows a battery of this apparatus as used in the Royal Testing Station at Berlin.

FIG. 63.—A Battery of Mechanical Briquette Formers in the Royal Testing Laboratory at Berlin, Germany.

STORAGE OF BRIQUETTES.

Environment During Setting.—It is common practice to place the cement briquettes, immediately after making, in a damp atmosphere and allow them to remain there for 24 hours. The purpose of this procedure is twofold:—first, to insure greater uniformity, and second to prevent the briquettes, especially those made from neat cement, from drying out too quickly and thus developing shrinkage cracks, thereby greatly lowering the strength. Uniformity is gained by reason of the fact that all the specimens acquire their set under precisely similar conditions of humidity, which cannot be controlled in the outer air, and which

TABLE XXXIV.—Showing the Effect of Variations in the Time of Storage in Damp Closet on the Strength of *Natural* Cement.
(From Sabin's "Cement and Concrete.")

Brand	Parts Crushed Quartz, 20–30 to 1 Cement	Age in Days, when Broken	Tensile Strength, Lbs. per Square Inch — Hours in Moist Air Before Immersion: 8	12	24	48	72	168
A	0	7	123	...	139	151	161	237
"	1	7	91	...	106	114	114	182
"	0	28	110	...	106	109	89	113
"	1	28	142	...	138	139	152	175
"	2	28	102	..	105	112	113	115
B	0	7	...	168	181	194	185	238
"	0	28	...	200	210	224	241	243
"	1	7	...	108	137	141	157	160
"	1	28	...	278	283	297	297	301
"	3	28	...	120	130	137	139	152

Each Result is Mean of 10 Tests.

NOTE.—Although these tests are made on natural cement alone, the action is typical of both Portland and natural cements.—The Author.

has been shown to have a great influence on the setting and hence early strength of the cement. Small ranges of temperature in the damp closet seem to affect the strength but little, but endeavor should be made to maintain an even temperature of as near 70° Fahr. as practicable.

The effect of duration of this treatment is shown in Table XXXIV., and generally is to increase the strength of the briquettes tested for short periods, especially those of neat cement; the difference, however, is slight and disappears after 2 or 3 months. The standard time for storage in damp closets has been fixed at 24 hours, largely as a matter of convenience.

The author has, however, shortened this time to 21 hours, the briquettes being made from 11 to 1 o'clock every day, and removed from the damp closet at 9 o'clock the following morning, thus giving time to mark the briquettes and clean the moulds before the briquettes for that day are ready to be made. The 24 hour neat briquettes are replaced in the damp closet for 3 hours.

Figure 64 is a sketch drawing of the damp closet used in the author's laboratory. It is made of $1\frac{1}{4}$-in. soapstone, except the

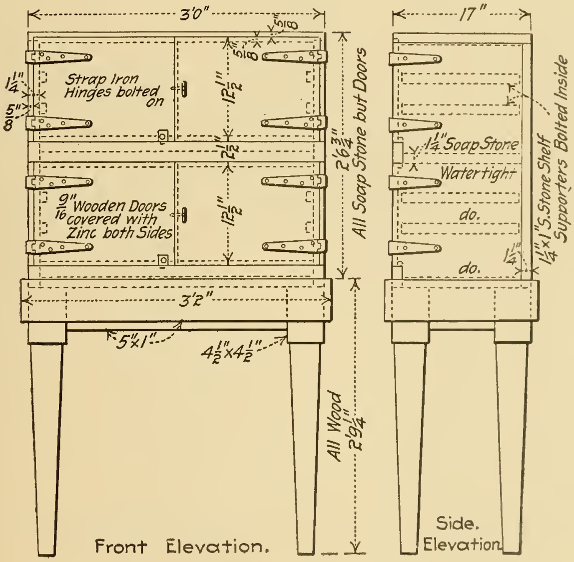


FIG. 64.—Sketch of Damp Closet Used in the Philadelphia Laboratories.

doors, which are of wood covered with zinc, and is made in two sections for the reason that it was found that if the height of the closet was excessive the humidity varied considerably between bottom and top. On the sides of each closet are fastened cleats, on the upper two of which rest the glass strips on which the neat briquettes are made, while the lowest pair of cleats supports a wooden rack, on which the moulds containing sand briquettes are placed on their sides. The water is placed in the bottom part of each section. Each section will accommodate 64 neat and 96 sand briquettes, and as many sections used as the

TABLE XXXV.—Comparison of the Strength of Portland Cement When Kept in Air and in Water.

(From a Large Number of Tests by the Author.)

Kept in	Mixture	Tensile Strength, Lbs. per Square Inch: 24 hours	7 days	28 days	2 mos.	3 mos.	4 mos.	6 mos.	1 year
Water	Neat	417	715	767	757	741	731	758	768
	1 : 1	361	593	692	690	680	679	684	696
	1 : 2	216	370	458	459	456	456	459	462
	1 : 3	105	210	302	309	310	312	310	309
	1 : 4	62	131	212	229	233	232	235	234
	1 : 5	38	82	154	186	196	198	195	198
Air	Neat	400	742	793	771	740	723	722	694
	1 : 1	349	625	702	696	690	694	692	690
	1 : 2	220	412	481	483	462	469	460	423
	1 : 3	101	239	357	342	363	360	371	352
	1 : 4	65	153	237	275	274	270	251	279
	1 : 5	46	124	206	249	252	237	237	239

TABLE XXXVI.—Showing the Relation Between the Strengths of Briquettes Kept in Air and in Water.

(From Falk's "Cements, Mortars and Concretes.")

Age	Tensile Strength in Lbs. per Square Inch: Neat Cement		1 Cement : 3 Sand		1 Cement : 5 Sand	
	Water	Air	Water	Air	Water	Air
7 days......	515	642	277	301	127	131
28 days......	658	651	350	438	180	247
84 days......	697	600	457	538	233	335
6 months ...	814	638	487	605	281	410
1 year......	765	575	550	703	271	442
2 years.....	838	507	503	650	270	408
Gauged with	23%		10.1%		9.5% water	

Note.—Each Value Based on 5 or 6 Briquettes.

TABLE XXXVII.—Effect of Temperature of Storage Water on the Tensile Strength of *Natural* Cement.

(From Sabin's "Cement and Concrete.")

No.	Brand	Parts Standard Sand to One of Cement, by Weight	Age in Days	Tensile Strength, Pounds per Square Inch when Immersed in Water of Temperature of—Degrees Fahr.: 38°	40°	50°	55°	60°	65°	70°	80°
1.	A	0	7	146	...	137	125	...	...	126	154
2.	"	0	14	144	...	131	125	131	150	168	208
3.	"	0	28	166	...	178	...	184	...	247	280
4.	"	1	7	83	...	88	84	89	98	97	121
5.	"	1	14	84	...	111	...	123	...	150	191
6.	"	1	28	96	...	156	187	...	221	243	288
7.	B	0	1	...	143	...	124	120	...	109	109
8.	"	0	7	...	204	201	...	183	...	193	186
9.	"	0	14	...	184	203	...	204	...	229	245
10.	"	0	28	...	221	245	...	254	...	281	303
11.	"	0	60	...	261	292	...	348	...	382	429
12.	C	1	7	...	134	140	...	150	...	154	158
13.	"	1	14	...	149	162	...	189	...	182	216
14.	"	1	28	...	198	223	...	250	...	281	296
15.	"	1	60	...	251	286	...	337	...	386	403
16.	"	3	14	...	50	58	...	69	...	73	100
17.	"	3	28	...	67	87	...	100	...	102	157
18.	"	3	60	...	104	127	...	147	...	194	231

Note.—The effect on the strength of Portland cement is similar, but not usually so well marked.—The Author.

volume of work requires. The expense of this closet may be decreased by making it entirely of wood lined with zinc; this will be quite good enough for any field laboratory, but will not give the satisfaction of the soapstone closet.

A device employed in the temporary laboratory of the Atlantic Avenue Improvement of the Long Island Railroad was to utilize stationary laundry tubs simply by fastening cleats on the sides to hold the glass strips and, although somewhat inconvenient, this appeared to be entirely satisfactory as regards results.

The use of some simple damp closet should always be required in even the most temporary of field laboratories. A damp cloth placed over the moulds may occasionally serve as a makeshift, but for regular work it is crude and inaccurate, unless given the most careful attention. If necessary to employ such a cloth, it should be so arranged that it never comes into contact with the briquettes and also that the ends of the cloth are placed in water, which prevents it from drying out quickly.

Storage of Briquettes.—After briquettes have hardened 24 hours in the damp closet, they are removed from the moulds and placed in water until ready for breaking. The chief reasons for storing in water rather than in air are that they are kept under conditions admitting of greater uniformity and that the effect of the presence of injurious elements is more marked. Neat briquettes kept in air generally are stronger in the early periods and weaker in the longer periods than those kept in water. Sand briquettes almost invariably are stronger in air (see Tables XXXV. and XXXVI.)

The temperature of the water (Table XXXVII.) slightly affects the strength of the briquettes, especially for the first period of 7 days. M. Alexandre* found that briquettes stored in water at normal temperatures gave higher values at 7 days than those at low temperatures, but that at 28 days the conditions were reversed for neat briquettes, and the sand briquettes gave almost equal values, while after 3 months no differences were apparent. The standard temperature is 70° Fahr.

The water should never show an acid reaction, nor be extremely alkaline. The gradual solution of certain salts from the briquettes soon makes the water strongly alkaline, and hence

*"Researches Experimentales sur les Mortiers Hydrauliques."

should either be kept running or frequently changed.* If running water is used the flow should be extremely slow to prevent any possible washing action. It has been claimed that running water tends to produce low results after a year or two, but tests now over three years old made by the author have shown no appreciable difference between running water and still water changed every two weeks.

The design of the storage tanks used in the Philadelphia

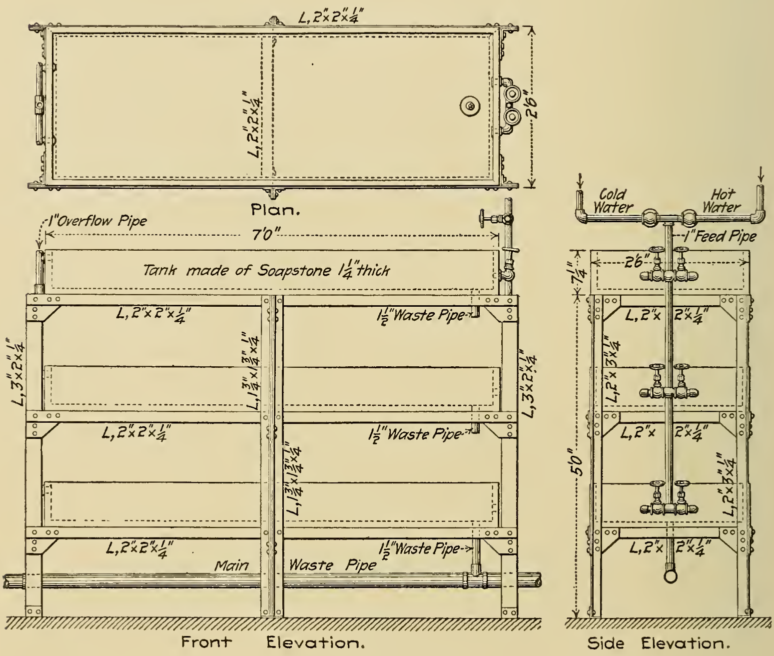


FIG. 65.—Sketch of Storage Tanks Used in the Philadelphia Laboratories.

Laboratories is shown in Fig. 65. They are made of 1¼-in. soapstone supported on a steel frame work. Hot and cold water are fed through separate pipes controlled by valves to maintain a uniform temperature, and the rate of flow is sufficient to change the water in each tank every hour. The capacity of each tank is about 1,400 briquettes, and the author has 12 of these in con-

*Mr. Sabin in "Cement and Concrete" states that the difference in strength of neat and sand briquettes of natural cement kept in fresh and stale water may amount to as much as 40 to 60 per cent.

stant use. It has been suggested that baffle plates placed at intervals along the tank so arranged that the water flows over and under every alternate plate would be of advantage in preventing the water from flowing across the top of the tank without running around the briquettes, but a long series of tests made by the author on briquettes stored at the top and bottom of the tanks showed no appreciable difference, thus proving such plates to be unnecessary.

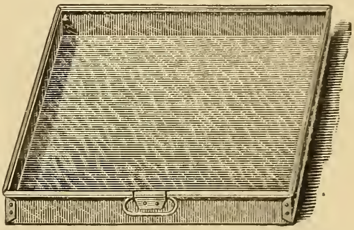


FIG. 66.—Pan for Storing Briquettes.

A more economical construction of these tanks, suitable for a temporary laboratory, may be made by building them of wood covered with zinc. If running hot and cold water is not obtainable they may be filled with a hose; a good method in this case is not to use the uppermost tank except for water, which is first fed into it, allowed to acquire the temperature of the room, and then fed to the briquettes below, thus avoiding any sudden chilling. For a still cheaper equipment, pans similar to those in Fig. 66 may be used. The water in these pans should be changed not less often than once a week, while larger tanks may be changed every two weeks.

Briquettes should always be placed in the water on their sides, never flat, so that the water may more readily circulate around them.

Marking Briquettes.—The author marks the briquettes with their numbers by means of a soft lead pencil, when removing them from the moulds, these marks remaining perfectly legible for at least 5 years.

Steel stamps are used in many laboratories, but it is necessary to place a thin strip of neat cement paste on the head of each mortar briquette to make the imprint visible. These stamps are applied immediately after the briquettes are made. They require considerably more time for marking than the lead pencil, and their use has no apparent advantage.

BREAKING THE BRIQUETTES.

Testing Machines.—For breaking the briquettes, machines are employed to apply the load and to measure the force necessary to cause rupture. Exclusive of the form and arrangement of the clips holding the briquettes, the requisites for a good machine are that it shall apply the load at a uniform rate starting from zero, that it shall be so arranged that the introduction of a systematic error is impossible, and that it shall be adaptable to rapid and at the same time accurate manipulation. The common types of these machines used in the United States may be divided into three classes: long lever, shot and spring balance. The long lever machines are probably the most accurate; the shot machines have the advantage of compactness and of operating without needing constant attention and hence are the most speedy; the spring balance machines are neither particularly rapid nor accurate, but are the cheapest and probably best suited to the engineer, who makes but a few tests at infrequent intervals.

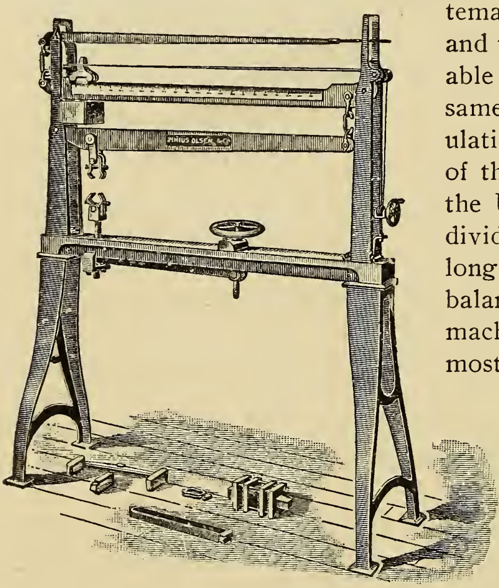


Fig. 67.—The Olsen Hand Cement Testing Machine.

The Olsen and Riehle standard machines are the most generally used of the long lever types. Figure 67 shows the Olsen hand power machine. The load is applied by means of a hand-wheel and lever, while the poise, moved by means of a hand-wheel and cord, registers the amount of stress applied. This design has many disadvantages, chief of which are that the stress is not uniformly applied, and second that the cord moving the poise is not in line with the knife edge of the beam and hence tends to pull it down, thus introducing a systematic error of no small magnitude. These objections have in a large measure been overcome in the power machine, shown in Fig. 68. In this arrangement the poise is mechanically driven along the beam at a uniform rate, while the beam is balanced by the wheel in the center of the frame which applies the load, and which can be operated by hand or by power, preferably the former. When the briquette ruptures, an electric contact is broken, stopping the poise instantly. By means of the step pulley the load may be applied at six different rates of speed.

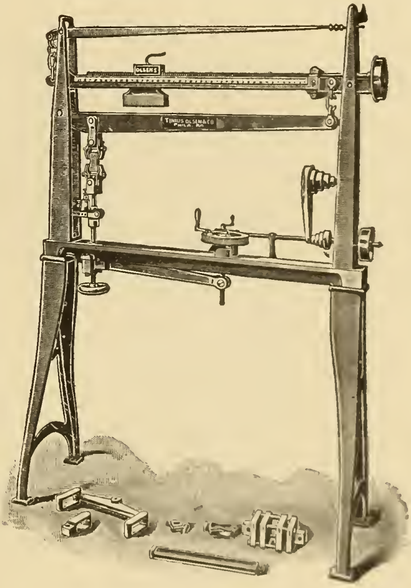


Fig. 68.—The Olsen Power-Driven Cement Testing Machine.

The Riehle power machine (Fig. 69) is built on lines somewhat similar to the Olsen, and is described by the makers as follows: "This machine is arranged for the stress to be applied to the specimen through belt and pulley, also by hand. Three speeds are obtained by shifting the belt on the cone pulley. A clutch controlled by the small handle starts and stops the movement of the screw; thus the belt can be run continuously. After the briquette is placed in position, this clutch is engaged and the screw applies the stress to the specimen. When the briquette breaks, the clutch is thrown out and the screw returned quickly to its original position by means of the hand-wheel under the clutch. The full capacity of the machine is registered on the beam, and no end weights or readjustment of the poise is necessary. The poise is operated by the hand-wheel near the beam; it is propelled by means of a screw and registers down to one pound. After the poise is moved out and the test completed, a lever which disengages the nut from the screw permits the operator to move the poise back to zero instantly."

Both of these machines are built of a capacity of 2,000 lbs., and both have attachments for making compression and transverse tests. The Riehle is also adapted for testing in torsion. While the general type of these two machines is very similar, it will be noticed that in the operation there is a radical difference in that the Olsen moves the poise at a uniform rate and balances it by hand application of the load, while the Riehle runs the screw applying the load at a uniform rate, and balances the poise by hand. Since there necessarily is, in a geared ma-

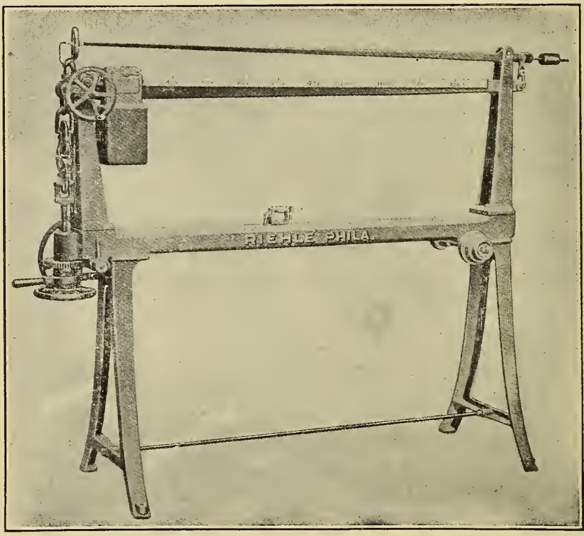


FIG. 69.—The Riehle Cement Testing Machine.

chine, considerable lost motion, and also on account of lost motion in the clips, especially when rubber surfaces are used, it will be found that although the belt running to the machine moves at a uniform speed, the stress applied by the latter method is not uniform, but that the rate increases with the stress, so that theoretically the former method is better. On the other hand, however, it is extremely difficult to apply the load in the Olsen machine at an absolutely uniform rate, especially in high testing briquettes, there being usually a more or less jerking motion which destroys the absolute continuity of the rate of stress. In practice, therefore, there is but little difference in the accuracy obtained, or in the efficiency of these two forms.

Since these long lever machines require power to operate them to the best advantage, occupy considerable space, and require more constant attention as well as some skill, a demand was created for a simple, automatic, compact device, requiring no power and little skill to manipulate. These conditions have been met in the "shot machines," of which the Fairbanks (Fig. 70) is the oldest and best known. The construction can be seen at once from the figure, and needs no further explanation. The method of operation given in the manufacturers' catalogue is as follows:

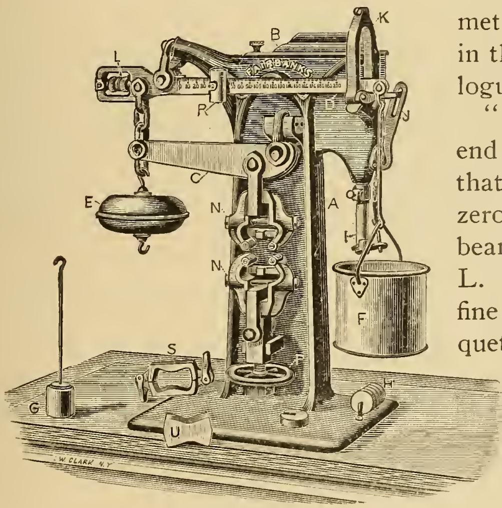


FIG. 70.—The Fairbanks Testing Machine.

"Hang the cup F on the end of the beam D. See that the poise R is at the zero mark and balance the beam by turning the ball L. Fill the hopper B with fine shot. Place the briquette in the clamps N-N, using great care to avoid eccentricity. Tighten the hand-wheel P sufficiently to cause the graduated beam D to rise to the stop K. Only enough pressure should be exerted to hold the beam firmly against the stop, not enough to transmit any strain to the specimen. Open the automatic valve J, so as to allow the shot to run into the cup F. At the point where the spout joins the reservoir will be noticed a small valve, by which the flow of shot may be regulated."

"When the briquette breaks, the beam D will drop and automatically close the valve J. Certain cements stretch or give to such an extent as to allow the beam to strike the valve before the specimen breaks. If this should occur, carefully raise the end of the beam with one hand until it again touches the stop K; with the other hand gently tighten the hand-wheel sufficiently to hold the beam in place, and again allow the shot to run. Under no circumstances should the wheel be tightened before the beam has been lifted against the stop, as such action invariably causes the specimen to break, rendering an accurate test impossible."

"After the specimen has broken, remove the cup, with its contents, hanging the counterpoise G in its place. Hang the cup F on the hook under the large ball E, and weigh the shot, using the poise R on the graduated beam D and the weights H on the counterpoise G. The result will show the number of pounds required to break the specimen."

The catalogue then goes on to say: "It has several times come to our notice that many users of these machines have been in the habit of applying an arbitrary strain by means of the hand-wheel, adding this strain to the actual result obtained in the proper manner. As one young man expressed it: 'We apply pressure with the hand-wheel equal to about four hundred pounds, as the other way is so slow.' This is so obviously unfair as to need no comment."

In spite of this assertion, however, the author believes the "young man" is in the right, for the reason that in following the method of the catalogue, the raising of the shot bucket entirely releases the load and brings the stress to zero, then suddenly applies the whole load again and increases it to the point of rupture, thus entirely destroying the continuity of the increase in stress. Moreover, this readjustment must take place when the briquette has nearly reached its ultimate strength, and even if the bucket is let down slowly, the sudden application of the load is liable to cause premature failure, so that evidently it is more accurate to apply an initial load of 200 or 300 pounds than to make this readjustment, which is exactly equivalent to applying an initial load of over 500 pounds. The proper method of operating this type of machine therefore is to apply such an initial load that on rupture the graduated lever will have lowered to a horizontal position, and is almost touching the valve.

This method, however, requires much experience and also a knowledge of about what value to expect from each briquette, so that even under the most favorable conditions it is not capable of precise determinations. This serious objection of necessary initial stress has been overcome in the "Improved Fairbanks" machine, shown in Fig. 71 described as follows:

"It is our regular cement testing machine equipped with a sub-base containing a worm and worm gear connected to an axis, which is threaded and passes up through the base and hand-wheel P into a block, and the latter connected to the lower clamp. The gear is actuated by the worm, the end of which is fitted to receive a key crank, passing through the front of the sub-base. A hook lever Y on the right hand end of the sub-base serves to disengage the worm from the gear, then the hand-wheel P may be used for rapid adjustment in returning the clamps to position to receive the next briquette."

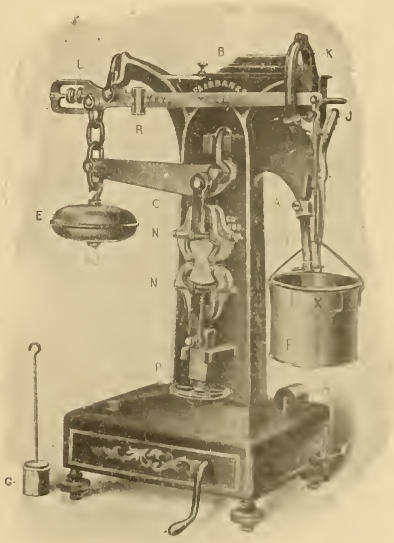


FIG. 71.—Improved Form of Fairbanks Cement Tester.

"In operation, the briquette is placed in the clamps, and adjustment made by the hand-wheel P until the indicators are in line. By means of the hook lever Y the worm is now engaged with the gear. The shot valve is then opened, allowing the shot to run into the bucket, and the crank is turned with sufficient speed to hold the beam in equilibrium until the briquette is broken."

A better method for practical operation is to adjust the lever as with the old machine to bear against the stop K, and then on those briquettes with which the lever drops too low, to engage the worm and float the lever with the crank. This may not be quite as accurate, but it avoids the constant attention of the operator, thus giving him time to note the value of the preceding test, and to get the next briquette ready, while the test is in progress, thus effecting great saving in the time of operation.

The only systematic error of any importance in the operation of this machine is due to the fact that after the rupture of the briquette a small amount of shot escapes from the valve before it can be closed, and also the shot falling through the air is weighed in the bucket, although not effective in producing rupture. The amount, however, is very small, may easily be determined by experiment, and can be either applied as a correction, or the beam so balanced that the error is compensated.

The "Falkenau-Sinclair" shot machine (Fig. 72), sold by Olsen & Co., is similar in type to the Fairbanks, although operating somewhat differently. *"The load is applied through a system of levers by means of the weight shown on the extreme right. Before starting a test this weight on the right is counterbalanced by shot held in the kettle on the left end of the same beam. To make the test the valve in the bottom of this kettle is opened, and as the shot escapes its equivalent of the weight on the right hand end of the beam acts on the briquette. The cut off of the shot is affected by the upper grip striking the horizontal arm which extends just above it, and thus releasing the curved arm carried on the spindle immediately to the left, this curved arm in turn striking the valve and closing it. The small hand-wheel for adjusting the lower grip is arranged so that it will automatically slip on the adjusting screw as soon as a predetermined load has been applied to the briquette."

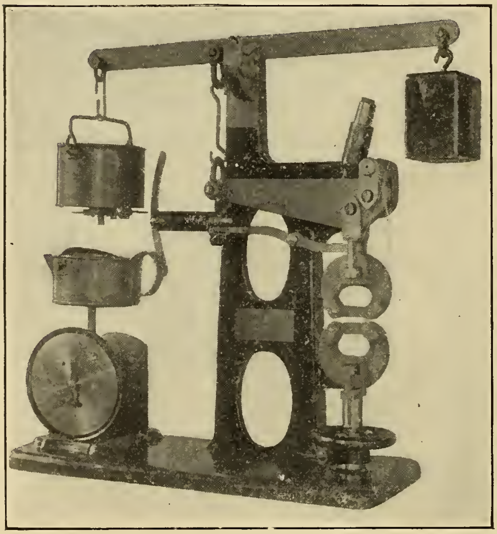


FIG. 72.—The Falkenau-Sinclair Cement Testing Machine.

The shot is weighed on a spring balance so graduated that the

*From manufacturers' circular.

load on the briquette is read directly. The advantages of this machine are the direct reading of the stress and the elimination of the errors due to the impact and weight of the falling column of shot; its disadvantages are the necessity of applying an initial load, the use of a spring balance which is less accurate and likely to introduce an undetected systematic error, and a rather complicated arrangement for closing off the flow of shot, liable easily to get out of order.

The Riehle machine (Fig. 73) eliminates the error due to initial loading by the use of a worm gear similar to that in the Improved Fairbanks, and, for stopping the flow of shot employs a piston valve, which is less easy to disarrange. Otherwise, it is very similar to the Falkenau-Sinclair.

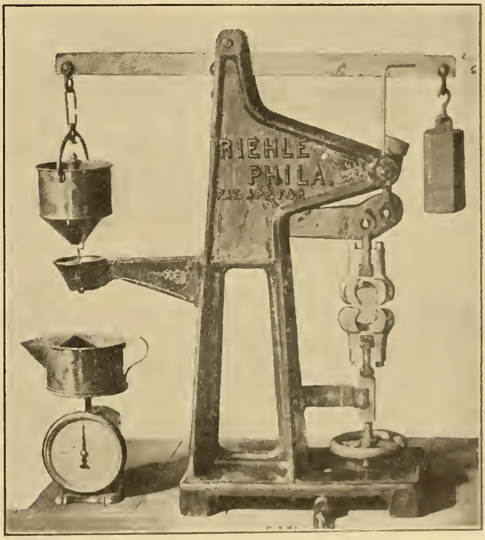


FIG. 73.—Shot Machine as Made by Riehle.

The capacity of the first two of these shot machines is 1,000 lbs., while the Riehle is made of both 1,000 and 2,000 lbs. capacity.

An example of the spring balance type of cement testing machine as made by Riehle is shown in Fig. 74. The stress is applied by turning the crank by hand and its amount indicated on the dial. The dial gauge has about an inch and a half of movement, and as it descends allows the wedge at the rear slide to drop and block the gauge and pointer from the shock of a sudden recoil at fracture, as well as leaving the register of the maximum load. The gauge is then relieved by means of the handle bar, the wedge withdrawn, and the pointer allowed to return to zero. The capacity is either 600 or 1,200 lbs. as desired. Olsen and others make machines practically similar.

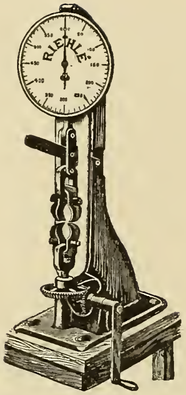


Fig. 74.—Cement Tester of the Spring Balance Type.

Several other forms of apparatus for testing briquettes have been designed, but none are in sufficiently general use to be considered. Of the forms of machine designed to test the tensile strength of cement using test specimens other than the normal briquette, the Johnson ring machine (Fig. 75) is one of the best known. The principle of operation is to burst an annular ring one inch high and half an inch thick by interior hydrostatic pressure. Such devices, however, although useful for certain classes of experimental work, are entirely impracticable for ordinary routine.

In either a permanent or a field laboratory, the shot machines will be found to be the most serviceable type for every day work, since they are quick to operate, have few parts to get out

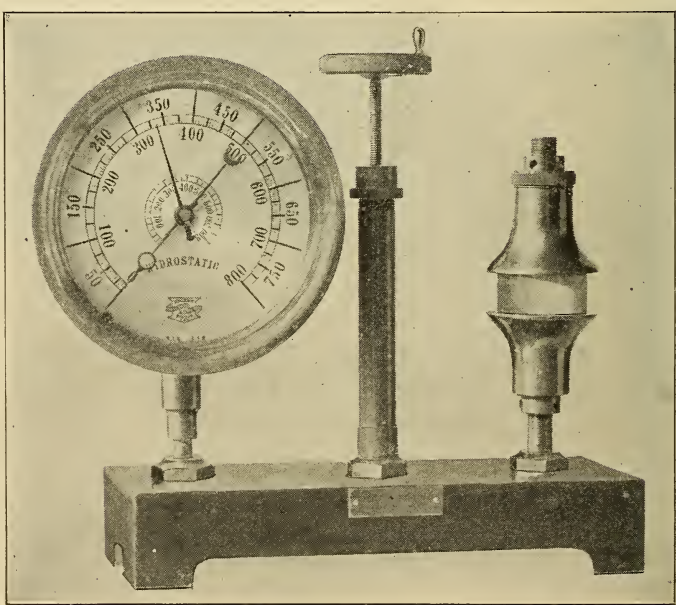


Fig. 75.—The Johnson Cement Testing Machine.

of order, and give sufficiently accurate results. For experimental research, however, the long lever machines are preferable on account of their greater accuracy. The attachments for compressive and transverse tests are also a valuable feature of the latter type. The author uses a long lever machine for experimental, and a shot machine for routine work, and believes this arrangement is the best for permanent laboratories. For the engineer making only occasional tests, the spring balance type is cheap, occupies but little space, and gives a fair approximation of the true values.

Form of Clip.—The standard form of clip (Fig. 76) recommended* by the Committee of the American Society of Civil Engineers has rigid bearing surfaces of brass, 1¼ ins. apart and ¼ in. wide, this last distance being shaped to fit the contour of the briquette. A bolt in the middle of the clip prevents the bearings from spreading. This rigid bearing is defective in that if there is any appreciable change in the volume of the briquette the contour of its sides and that of the bearings no longer agree and the bearing reduces to a line instead of an area, thus creating a greater tendency to breaking in the clip. To overcome this, many automatically adjustable bearing devices have been proposed, including roller bearings, conical bearings, adjustable plate bearings, pin-connected clips and several others. These devices when in good working order generally reduce slightly the proportion of clip breaks, but give

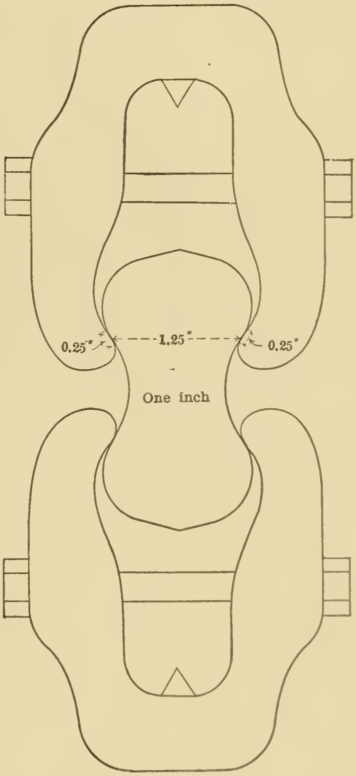


Fig. 76.—The Standard Form of Clip Recommended by the A. S. C. E. Committee.

*See Appendix A.

strength values no greater than with the rigid form. The great objection to these adjustable bearings is the difficulty in keeping them clean and working smoothly, and if a small piece of cement becomes wedged in them, thus preventing freedom of motion, they are much worse than the rigid bearing. Roller bearings are especially objectionable in this particular, being easily clogged and then wearing flat.

The use of cushioned clips has been frequently attempted by inserting strips of rubber, blotting paper or tin-foil, between the briquette and the bearing, but, although effective in preventing clip breaks, they give much lower strength values. Sabin* states that in a series of tests cushioned clips gave but 86 per cent. of the strength of briquettes tested in bare clips. Tests by the author gave a smaller difference, but still an appreciable lowering of the strength. In routine work these cushions are annoying and unsatisfactory.

The reason that briquettes break in the clips must be that cross-strains are developed, which cause premature failure, and furthermore, it is evident that the strength at the least section of the briquette should be greater than the result obtained from a clip break, because a stress of that amount has not produced failure. Nevertheless, it is a fact that breaks in the clips average a greater strength than those breaking in the least section. Comparison of these values by the author on over 1,000 briquettes showed the clip breaks to average about 4% greater than those breaking normally. Sabin found this difference to average $3\frac{1}{2}$%, and says "this result is easily accounted for by saying that some of the briquettes that broke in the small section were made to do so by the cross-strain introduced by imperfect adjustment in the clips." This reason, however, seems scarcely sufficient.

The standard form of clip will give about 5 to 10 per cent. of clip breaks for neat cement briquettes, and almost none for sand briquettes. It is far the quickest and most convenient to operate, never gets out of order and will be found the most satisfactory for ordinary routine. The use of strips across the backs of the clips for purposes of adjustment has been found inconvenient, better and quicker adjustment being made on clips open on both faces.

*"Cement and Concrete," by L. C. Sabin.

Great care should be exercised to see that the briquettes are properly centered and the bearings immediately over each other. Johnson* states that an eccentricity of 1/16-in. may reduce the tensile strength by as much as 15 or 20 per cent.

Rate of Stress.—The more rapidly the load is applied to a cement briquette, as with all other materials, the greater will be the results obtained. The diagram (Fig. 77) gives the results of tests made by Henry Faija in 1883, while Fig. 78 represents a short series made by the author. The trend of both diagrams is seen to be similar. Both of these diagrams were based on tests of neat Portland cement briquettes only, but Table XXXVIII shows that the same law applies to briquettes of all compositions. The standard rate for many years has been 400 lbs. per minute, but the recent Committee of the American Society of Civil Engineers has increased this rate of applying the load to 600 lbs., which commends itself both for increased rapidity in testing, and also that, as shown by the diagrams, small variations from the standard have less effect at the higher rate. Any testing machine should be carefully set and made to operate regularly at this fixed speed. In a long lever machine it must be remembered that it is the poise that must move uniformly, not the wheel applying the load.

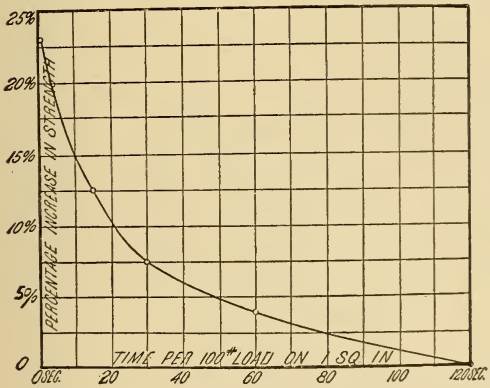


FIG. 77.—Diagram Illustrating the Effect of Rate of Application of the Load on the Tensile Strength of Cement. (From Faija's Tests.)

Wet Briquettes.—Cement briquettes must always be broken as soon as they are removed from the tanks and before they have commenced to dry out. Experiments have shown that this first drying out greatly lowers the strength, especially of neat briquettes, and that half an hour's time may appreciably affect the results.

*"Materials of Construction," by J. B. Johnson.

No more than 5 neat, nor 10 sand briquettes, should be taken at once from the tanks, and placed in air near the testing machine. If the storage tanks are at some distance from the ma-

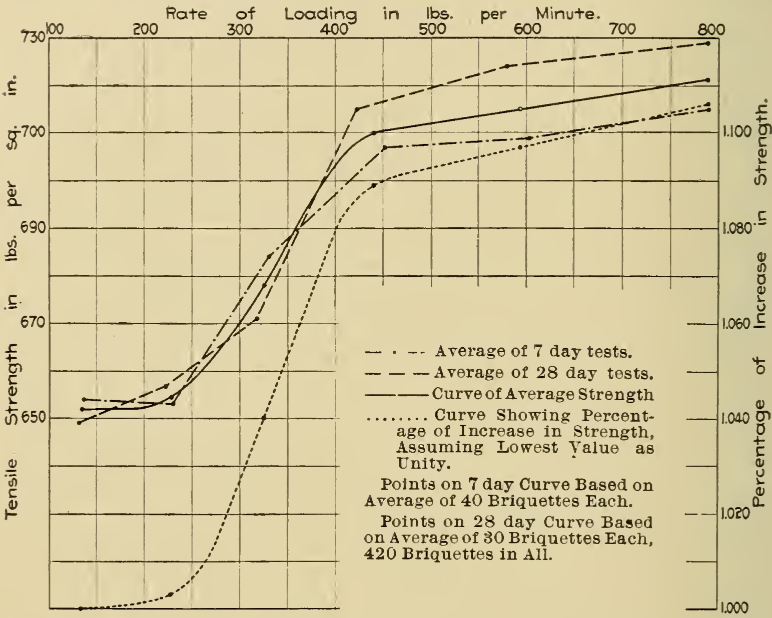


FIG. 78.—Diagram Illustrating the Effect of Rate of Application of Stress on the Tensile Strength of Cement. (From Tests by the Author.)

chine, the briquettes ready for test should be kept in a pan filled with water placed conveniently near from which they can be taken 3 or 4 at a time.

Number of Briquettes.—The number of briquettes to be broken

TABLE XXXVIII.—Effect of Rate of Applying the Stress, on Tensile Strength.
(From Sabin's "Cement and Concrete.")

Cement	Proportions	Age	Tensile Strength, Pounds per Square Inch, for Stress, Applied at—Pounds per Minute.				
			100	300	500	700	900
Portland	Neat Cement	7 and 14 days	453	485	521	520	528
"	" "	3 months	...	590	617	622	640
"	1 : 2	3 "	445	467	487	507	510
Natural	Neat Cement	7 days	150	169	186	...	202
"	" "	3 months	309	351	363	378	390
"	1 : 2	3 "	255	299	327	329	354

at each period depends upon the importance of the test, the accuracy desired, and the skill of the operators. In ordinary routine, the author makes but 8 neat and 6 sand briquettes from each sample, breaking 2 neat briquettes at 1, 7 and 28 days, and two sand briquettes at 7 and 28 days. If the average of these two values meets the requirements of the specification, or falls far below them, no other tests are made, but if the average is but slightly below, especially if one test is over and one under, one, or if necessary, both of the remaining two briquettes are tested to corroborate one of the two values. If the additional briquettes are not thus needed they are stored and broken at later periods for the accumulation of data. The testing of so few briquettes is, however, only possible where the operators have attained a high degree of skill and accuracy, and is not advised for the ordinary laboratory. Generally, reception tests should be made on 4 or 5 briquettes for each period, which may be reduced to 3 or 4, as the accuracy of the operators increases. For experimental work, or where especial accuracy is desired, as in a possible case of litigation, at least 10 briquettes should be tested for each period. If the number of briquettes to be made from any one sample is too great to be made from one mixing and moulding, the briquettes for each period should be taken equally from each moulding, that is, if 2 mouldings of 8 briquettes each are made, and 4 briquettes are to be broken at 7 days, 2 should be taken from the first moulding, and 2 from the second. If gang moulds are used, and only one mixing is made, then briquettes for any one period should be taken from different gangs and not from the same one.

Average Values.—The result of the test is the arithmetical mean or average of the strength of the individual briquettes. Some writers claim that the highest value and not the average should be taken as the result, but this is manifestly inaccurate for the reason that the determination is not made to ascertain the greatest strength that the cement can develop, but the strength it will attain when treated in accordance with certain fixed conditions, which is only represented by the average. It is, of course, true that most of the irregularities introduced by careless manipulation tend to lower, rather than increase, the strength, but it, nevertheless, may well happen that the highest value may also be the result of some abnormal condition and not be indicative of the true strength.

Accuracy.—The accuracy of a test depends upon the skill of the operators in making uniform briquettes and upon the number of individual values from which the average is computed. The accuracy of a series of tests is determined by computation of the probable error* of a single determination and of the mean. A skilled operator should always work with a probable error of not over 4 per cent. for a single briquette. Any testing in which the probable error of a single result exceeds 7 or 8 per cent. is very inaccurate and is indicative of either gross carelessness, or of the use of a poor method. An approximate method of stating the accuracy of a series of tests is by means of the average departure from the mean which is the arithmetical mean of the individual errors. In cases where a long series of tests has been made to obtain a single result a method sometimes employed is to discard all values whose departure from the mean exceeds say 10 per cent., and then to average the remaining values for the final result.

Thus supposing the 20 tests given in Table XXXIX. were made to determine the strength of sand briquettes at 28 days, all of them, apparently, being equally well made and broken. The mean value is 281 pounds; the probable error of one result is 13.2 pounds, or 4.7%, of the mean, while that of the mean is 3.0 pounds, or 1.1%; the average error is 15.6 pounds, or 5.5%. The probable error of the mean result is expressed by stating its amount with a plus or minus sign after the average. Thus to state that a cement has a strength of 281 pounds gives no indication of the accuracy of determination, but if it is stated that 20 tests gave a result of 281.0 ± 3.0 pounds, the amount of dependence that can be placed on any value is positively

* The probable error is an error of such a value that the probability of its being exceeded is equal to the probability of its not being exceeded. From the principles of the method of least squares, the probable error of a single determination, in a series of determinations of equal weight, is computed to be

$$E_s = 0.6745 \sqrt{\frac{\Sigma \Delta^2}{n-1}}$$

in which Δ = the difference between any one determination and the mean value of the series, and n = the number of determinations. The probable error of the arithmetical mean of the series is

$$E_m = 0.6745 \sqrt{\frac{\Sigma \Delta^2}{n(n-1)}} = \frac{E_s}{\sqrt{n}}$$

known. The method of correction by dropping values whose error exceeds ten per cent. of the mean is also shown in Table XXXIX., by which it is seen that the average is changed from 281.0 ± 3.0 to 282.9 ± 2.3 pounds. This series of tests is of but indifferent accuracy.

TABLE XXXIX.—Illustration of the Method of Computing Probable and Average Error, and of Correcting a Series of Determinations.

	Original			Corrected		
No.	Value	Error	Square of Error	Value	Error	Square of Error
1.	271	10	100	271	12	144
2.	277	4	16	277	6	36
3.	266	15	225	266	17	289
4.	286	5	25	286	3	9
5.	284	3	9	284	1	1
6.	252	29	841	...	..	...
7.	307	26	676	307	24	576
8.	271	10	100	271	12	144
9.	298	17	289	298	15	225
10.	251	30	900	...	..	...
11.	279	2	4	279	4	16
12.	272	9	81	272	11	121
13.	248	33	1,089	...	..	...
14.	316	35	1,225	...	..	...
15.	267	14	196	267	16	256
16.	285	4	16	285	2	4
17.	282	1	1	282	1	1
18.	303	22	484	303	20	400
19.	295	14	196	295	12	144
20.	310	29	841	...	..	...
Total	5,620	312	7,314	4,243	156	2,366

	Lbs.	% of Mean
Original:—		
Mean	281.0	...
Probable error of one result	13.2	4.7
" " " mean	3.0	1.1
Average error	15.6	5.5
Total range	68.0	24.2
Corrected:—		
Mean	282.9	...
Probable error of one result	8.8	3.1
" " " mean	2.3	0.8
Average error	10.4	3.7
Total range	41.0	14.5

Of course, this method of stating accuracy and correcting results applies more to experimental work or other cases where extreme accuracy is desired. In ordinary routine the range in the values of 3 or 4 briquettes should not exceed 10%, while the probable error of a single value should not average over 3½ or 4 per cent.

The Tensile Strength of Cement.—On account of the many irregularities in the testing of briquettes, as well as the complex influences, both interior and exterior, operating upon the cement, the curve of hardening of any one series of tests will present many apparent anomalies. The average values obtained in the Philadelphia Laboratories up to a period of one year are shown in Fig. 79, the curves of the neat and 1:3 sand briquettes being based upon an average of over 150,000 briquettes, while the other curves are based upon 300 to 500 values each. The sag in the curve of the neat briquettes is plainly evident and the probable explanation of this condition has already been given.* After a period of one or two years the

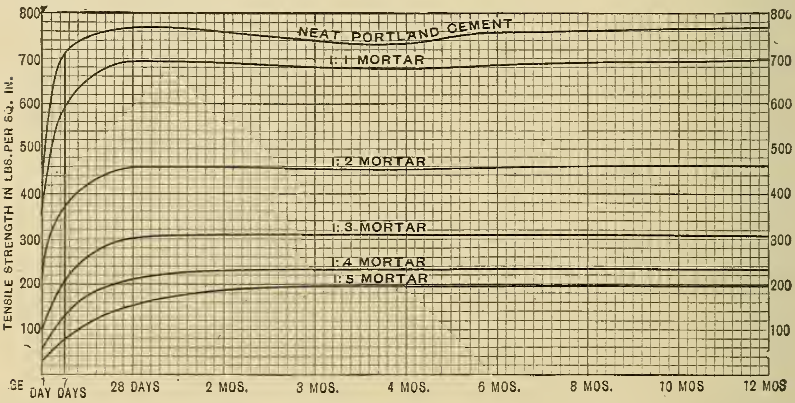


FIG. 79.—The Average Strength of Portland Cement. (From Tests by the Author.)

values again begin to show retrogression from which there is no apparent recovery. This, apparently, is due to a change in the structure of the cement, for up to that time the fracture appears dull and earthy, while later it becomes distinctly glassy and brittle, thus making more decided the effect of the irregular crushing and cross-breaking strains that act in the least section of the briquette. It is probable, therefore, that the pure tensile strength is never developed after this change in structure takes place, and hence the results show an apparent weakening although the real structural value of the material is in no wise affected by the alteration. This action also is developed to a far greater extent in the small mass of a briquette than could take place in the large volumes of construction work,

*See page 103.

and although often existing in apparently alarming proportions, it need never cause anxiety as to the safety of the structure unless accompanied by a noticeable change in volume or by actual disintegration.

There have been many formulas proposed by which the strength of cement at any period may be computed, if the values at 7 and 28 days are known, but they all assume that the rate of hardening progresses according to some definite law, and thus fall into such positive error that they fall little short of being absurd.

It should be stated that both the first sag and final retrogression in the curve of neat tensile strength is much more marked in the case of cements manufactured by the rotary kiln process than in those made in stationary kilns, although there is but little, if any, difference in the structural value of the two classes of material. The difference in briquettes of sand mortar is less apparent.

Interpretation of Results.—Specifications for the tensile strength of cement usually stipulate merely that the material pass a minimum strength requirement at 7 and at 28 days, and the requirements, moreover, are so easily met that only a decidedly inferior cement will fail to pass them. It must not be understood, however, that the specification requirements should be raised, since many old and well seasoned cements which make the best material for service might then be rejected. The proper grounds for the judgment of the tests of tensile strength are four in number:—that both neat and sand briquettes shall pass a minimum specification at 7 and at 28 days; that the neat value at 7 days shall not be excessively great; that there shall be no retrogression in the neat strength between 7 and 28 days; and that the strength of the sand briquettes between these periods shall increase at least 10 or 15 per cent. It must, moreover, be remembered that the sand test is the true criterion of strength, and no cement failing in these tests should be accepted even if the neat results are excellent. If the conditions are reversed, however, the sand tests passing and the neat failing, it may be justifiable to permit the use of the material, provided there is no accompanying indication of unsoundness.

The reason that the strength of cement shall satisfy a minimum requirement is obvious. The objection to a high neat test at seven days is that it usually indicates an over-limed cement, which is practically certain to develop a decided retrogression in 28 days, and is also more liable to unsoundness. An abnormal amount of sulphate of lime may also produce a similar effect. Portland cement tested neat in accordance with the method given and developing a strength in 7 days of over 850 pounds should be looked upon with suspicion and generally ought to be held for the 28-day test before allowing it to be used. Cement showing a retrogression in the strength of neat briquettes between 7 and 28 days is not necessarily of poor quality, but it may be considered as inferior to those giving a good increase. On cements testing below 750 or 800 pounds at 7 days, the lower the 7-day results, the more serious becomes any subsequent falling off in strength. If testing below 700 pounds at 7 days, retrogression should mean rejection. In general, the greater the increase in strength between specification periods, the greater will be the strength ultimately attained. Thus one testing 550 and 700 pounds at 7 and 28 days is usually preferable to one testing 750 and 800.

Cements failing to pass the sand requirements, or those not increasing in the sand strength, should not be accepted. Retrogression in sand strength is indicative, in the majority of cases, of ultimate complete failure.

A fair strength specification for Portland cement tested in accordance with the given method is 500 pounds for 7, and 600 for 28 days, for neat briquettes, while those made of 1 part cement to 3 parts standard quartz should exceed 170 and 240 pounds at the same periods. If Ottawa sand is used, the sand requirements should be increased to 200 and 280 pounds. On these figures material passing the 7-day tests and failing at 28 days is unsafe, while one failing at 7 and passing at 28 may be accepted. Additional security may be obtained by specifying a maximum neat strength at 7 days (from 850 to 900 pounds), and an increase of 10 per cent. in the sand strength between 7 and 28 days.

One other point that must always be borne in mind is that cement has no absolute strength, but the strength is that developed by a certain process of manipulation; if, therefore, the process varies, the results will also. For this reason the method to be employed in obtaining the results should be a feature of every strength specification. In many cases, after the rejection of a shipment, those furnishing the material have tests made by private laboratories and apparently disprove the original tests, but such tests deserve no consideration whatever, unless it be proven that the methods employed were identical in both cases and that both conformed to that stipulated in the specifications. An experienced operator may obtain almost any result from any cement by changes in the manipulation.

The following rules for the acceptance or rejection of material on the results of the tensile test represent safe and conservative practice:

At 7 days: Reject on a decidedly low sand strength.
Hold for 28 days on low or excessively high neat strength, or a sand strength barely failing to pass requirements.

At 28 days: Reject on failure in either neat or sand strength.
Reject on retrogression in sand strength, even if passing the 28-day requirements.
Reject on retrogression in neat strength if there is any other indication of poor quality, or if the 7-day test is low—otherwise accept.
Accept if failing slightly in either neat or sand at 7 days and passing at 28 days.

CHAPTER X.

SOUNDNESS.*

Definition.—The soundness of cement may be defined as that property which resists any force tending to cause disintegration or lack of permanency in the structure, and since, if such disintegration occurs, it is usually accompanied by change of volume, a sound cement is frequently termed "volume constant." This determination of "constancy of volume" or "soundness" is unquestionably the most important phase of the testing of cement, for although a sample may pass all the other tests with ease, if it is unsound, and will eventually disintegrate on the work, it is evidently worse than worthless for constructive purposes.

Causes of Unsoundness.—The most important factor operating in a cement to cause unsoundness is an excess of lime, either free or loosely combined, which has not had opportunity to have become sufficiently hydrated. The presence of this lime may be due to incorrect proportioning, to insufficient grinding of the raw materials, to underburning, to lack of seasoning, or to coarseness of the finished cement which prevents perfect hydration. Excess of magnesia or the alkalies and the presence of sulphides are also sometimes responsible for disintegration, while the presence of sulphate of lime may act in either direction, occasionally causing unsoundness, but generally tending to make good an otherwise unsound cement, at least so far as laboratory tests are concerned.

Although an excess of uncombined or loosely combined lime is generally conceded to be the most potent factor in causing unsoundness, it is nevertheless impossible to tell by any known method of chemical analysis just what proportion of the total lime present exists in this dangerous condition, so that unless any injurious constituent is present in gross excess, an-

*Much of this chapter is quoted from a paper on "Soundness Tests of Portland Cement," read by the author before the American Society for Testing Materials. July, 3, 1903.

alysis gives no indication as to the soundness of the material.*

Excluding, therefore, the effect of composition, which is usually indeterminable, the conditions most affecting the soundness of cement are its age or seasoning, and its fineness.

Effect of Age.—The property of a cement most affecting the results of the tests for soundness is its age or the amount of seasoning it has undergone. Almost every cement, no matter how well proportioned and burned, will contain at least a small amount of free or loosely combined lime, which will often cause unsoundness if used or tested at once. This lime, however, will hydrate in a very short time on exposure to the air, thus rendering it inert and preventing any expansive action. It will,

TABLE XL.—Effect of Age of Cement on Results of Boiling Test.
(Tests by the Author.)

Age of Cement When Tested	Tensile Strength — Neat 1 day	Neat 7 days	Neat 28 days	1 : 3 Sand 7 days	1 : 3 Sand 28 days	Normal Pat Tests 28 Days in Air	Normal Pat Tests 28 Days in Water	Boiling Test
1 week	550	765	762	171	225	Curled and soft.	Slightly checked.	Partly disintegrated.
2 weeks	548	767	771	170	246	Slightly curled.	Slightly curled.	Checked and cracked.
3 "	492	718	763	182	244	"O. K."	"O. K."	Slightly checked.
5 "	427	692	747	183	249	"O. K."	"O. K."	Sound.

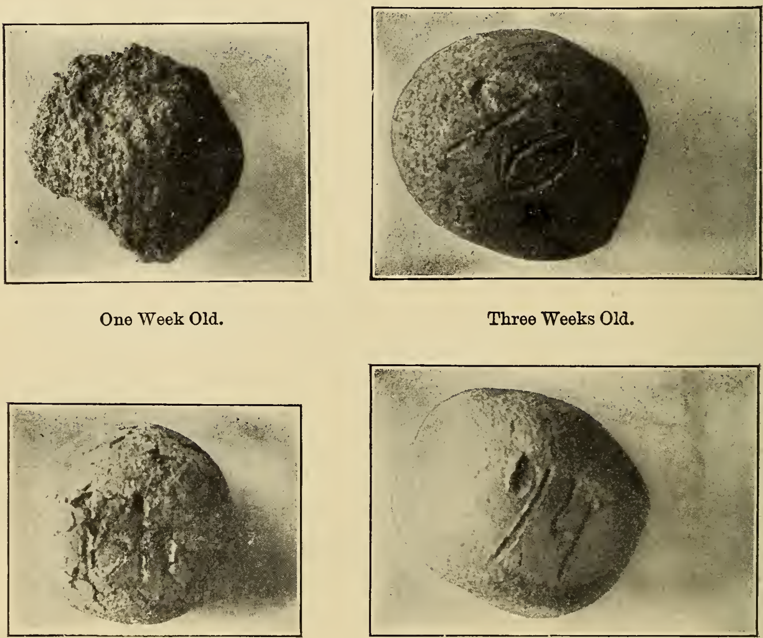
therefore, be found that, in a large majority of cases, if a cement failing in the normal or accelerated tests be stored for two or three weeks, this unsoundness will disappear, and the cement pass the tests with ease. A typical case of this is shown in Table XL., the specimens on which the boiling test was made being also shown in Fig. 80. It will be noticed that in this instance the cement has been made thoroughly sound by a seasoning of five weeks. The early strength values of the neat tests are also seen to fall off decidedly, while the sand tests generally show a slight increase.

Effect of Fineness.—Coarseness of grinding is also a frequent cause of unsoundness for the reason that the larger particles are not readily susceptible to hydration, and contain for a long period of time expansive elements, which very rapidly develop

*For further discussion on this point see Chapter XI.

a disintegrating action when treated in the accelerated tests, and even in the normal tests often induce failure. A study of the tests given in Table XLI., the boiling test specimens of which are shown in Fig. 81, will clearly show that failure in this instance was caused by the presence of expansives in the coarser particles.

Methods of Determining Soundness.—The soundness of cement is customarily determined in one or more of the three following



One Week Old. Three Weeks Old.

Two Weeks Old. Five Weeks Old.

Fig. 80.—Illustrating the Effect of Age on Soundness. (See Table XL.)

ways:—by direct measurement of the change in volume; by observation of specimens kept in a normal environment—called "normal" tests; by observation of specimens so treated by the action of heat or chemical salts that any disintegrating action is hastened—called "accelerated" tests.

Measurements of Expansion.—Soundness was often tested by this method some years ago, but at present it has been vir-

No. 1.—Cement as Received (very coarse).

No. 4.—Cement Finely Ground (tested one week later than No. 1).

No. 2.—Cement as Received (sifted to pass No. 200 sieve).

No. 5.—Cement Finely Ground (tested two weeks later than No. 1).

No. 3.—Cement Finely Ground (tested same time as No. 1).

No. 6.—Cement as Received (tested two weeks later than No. 1).

FIG. 81.—Illustrating the Effect of Fineness on the Boiling Test. (See Table XII.)

tually abandoned in the United States, although still employed considerably abroad. It has been definitely shown that even an apparently high expansion or contraction is not necessarily indicative of disintegration, while on the other hand cases have frequently been observed in which a cement has remained sound and without appreciable change in volume for several months.

then suddenly begun to disintegrate and finally failed entirely. In construction work, an expanding cement is deemed beneficial by many prominent engineers, as compensating in a measure for settlement. Small specimens of cement kept in the laboratory will usually show contraction when kept in air, and expansion when kept in water.

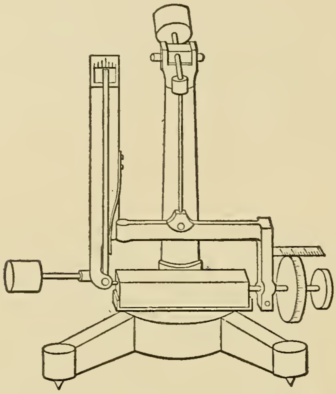


FIG. 82.—Calipers for Measuring Expansion, According to Bauschinger.

For measuring the amount of change in volume, the Bauschinger apparatus, shown in Fig. 82, is one of those most generally used. The principles of this apparatus can be plainly seen from the figure. The bars of cement are about 5 square centimeters in section and 10 centimeters in length, and have small plates embedded in the ends to serve as bearing points for the micrometer screw, which will indicate a change in length of .0005 centimeters. The vertical needle and spring on the left of the figure insure a uniform pressure of the screw against the bearing plate.

TABLE XLI.—Effect of Fineness of Cement on Results of Boiling Test. (Tests by the Author.)

Condition of Cement	Fineness			Boiling Test
	No. 50	No. 100	No. 200	
As received................	0.5	13.2	33.4	Badly checked and cracked.
Same cement sifted.........	0.0	0.0	0.0	Sound.
Same cement ground	0.0	0.6	3.0	Checked and cracked.
Ground cement, 1 week later	0.0	0.6	3.0	Very slightly checked.
" " 2 " "	0.0	0.6	3.0	Sound.
As received 2 " "	0.5	13.2	33.4	Checked and cracked.

The Le Chatelier apparatus (Fig. 83) is also infrequently employed for this purpose and is said to be more easily operated than any of the other forms. It is described by Le Chatelier as follows: "A much more simple and yet sufficiently precise measurement of the expansion can be made by letting the cement harden in cylindrical moulds of a diameter equal to their height, constructed of metal, slit along the generatrix and provided on each side of the slit with two long needles, which serve to magnify any widening of the slit. The widening is equal to the enlargement, not of the diameter, but of the circumference of the cylinder of cement."

This apparatus may be employed not only on specimens kept at normal temperatures, but also on specimens which have undergone some form of accelerated test. The method of making this test, recommended by the Engineering Standards Committee in the British Standard Specifications,* is as follows: "The apparatus for conducting the Le Chatelier test consists of a small split cylinder of spring brass or other suitable metal of 0.5 millimeters in thickness, 30 millimeters internal diameter, and 30 millimeters high, forming the mould, to which on either side of the split are attached two indicators 165 millimeters long from the center of the cylinder, with pointed ends."

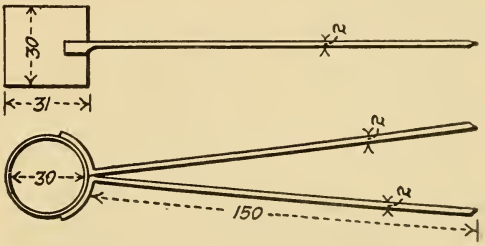


Fig. 83.—The LeChatelier Tongs.

"In conducting the test, the mould is to be placed upon a small piece of glass and filled with cement gauged in the usual way, care being taken to keep the edges of the moulds gently together while this operation is being performed. The mould is then covered with another glass plate, a small weight is placed on this and the mould is immediately placed in water at 58 to 64 degrees Fahr., and left there for 24 hours."

"The distance separating the indicator points is then measured, and the mould placed in cold water, which is brought to a boiling point in 15 to 30 minutes, and kept boiling for six hours. After cooling, the distance between the points is again measured, the difference between the two measurements representing the expansion of the cement."

While there exist many other forms of apparatus for making this determination, they all are more or less similar in principle to those given, and since this test is seldom, if ever, required in this country, it will not be considered further. Possibly allusion should be made to the once popular "lamp-chim-

*See Appendix E.

ney" test, which was based upon the same idea, and consisted of filling a lamp chimney with a thin paste of cement, cracking of the chimney in hardening showing expansion, and being considered a failure. This test, however, was so crude, and the inferences drawn from it apt to be so erroneous that it has been almost entirely abandoned.

Normal Tests.—Normal tests consist of making, from pastes of neat cement, pats having thin edges, being thus more susceptible to any disintegration, keeping them in air and in water under normal conditions and observing them from time to time, to see whether they remain hard, sound and straight. This, undoubtedly, is a perfectly fair test, with a possible exception, in that a neat cement is always more liable to disintegrate than a sand mortar, and hence a cement may occasionally fail in the normal tests, while the mortar in which the cement is used may be perfectly sound. Generally, however, failure in the normal tests is indicative of unfitness for use.

The common form of specimen for these tests is a circular pat, 3 or 4 inches in diameter, about $\frac{3}{8}$ to $\frac{1}{2}$-inch thick at the center, and tapering to a thin edge on the circumference. Sometimes the specimens are made in the form of a wedge, with a thin edge on only one side. These pats should be made of neat cement of normal consistency and should be kept in either air or water at a temperature of as near 70 degrees Fahr. as practicable, although small variations from either normal consistency or normal temperatures fortunately seem to exert but little influence on the results. A most important point, however, always to be observed, is that the pats, as soon as made, be placed in a damp closet or covered with a damp cloth until they have entirely hardened (best for 24 hours), since otherwise, if allowed to dry out too quickly, they may show shrinkage cracks which by a novice might be mistaken for failure. It is also common practice to mould the pat on a small square of polished glass, and to allow it to remain attached to this glass during the entire period of the test.

The specification test is generally for 28 days, although pats should be kept for a much longer time if reliable data concerning them are desired, and it is good practice to examine these pats at intervals of 3, 7, 14 and 28 days from the date of making, and then at such intervals as may be desired. To thoroughly examine a pat, it should be ascertained (1) whether it has left the glass, (2) if it has left the glass, whether it is straight or curved, (3) whether it has developed cracks due to shrinkage, expansion, or disintegration, (4) whether it is blotched, (5) whether the glass is cracked. For examination of the curvature a small steel straight-edge is convenient.

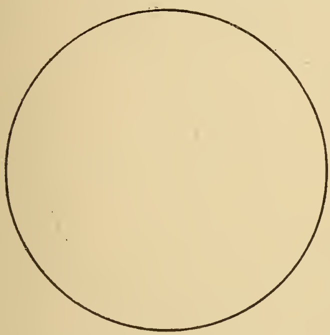


Fig. 84.—Normal Pat.

Pats kept in water should be straight, free from cracks, and not blotched. Pats in air should not show disintegration cracks, should not be excessively curled, nor blotched. Cracking the glass in the water pat, expansion cracks and slight curvature in the air pat, and leaving the glass in either air or water are not considered to be indicative of injurious properties. About 30% of the water and 70% of the air pats leave the glass in seven days' time.

Since a person with limited experience in cement testing often desires to make and test normal pats, the following diagrams are given to illustrate the common forms of failure in these tests:

Figure 84 represents a normal pat in good condition.

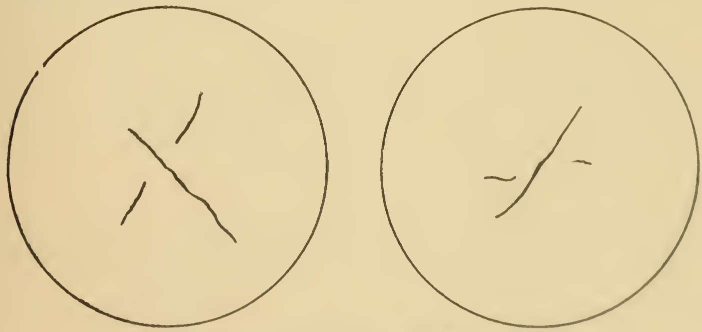


Fig. 85.—Shrinkage Cracks.

Figure 85 represents shrinkage cracks. These are due ordinarily to the use of too wet a mixture, or to too quick drying out. If the pats are left exposed to dry air during setting, these cracks frequently develop. Shrinkage cracks ordinarily, therefore, indicate only a lack of care in manipulation, and not dangerous qualities of the material.

FIG. 86.—Expansion Cracks.

Figure 86 illustrates cracks caused by expansion or contraction. In the air pats these cracks are developed in nine-tenths of those adhering to the glass, and unless very decidedly marked, are not dangerous. If existing in the water pats, however, it usually indicates an inadmissible proportion of expansive elements.

Figure 87 represents pats curling away from the glass, but still adhering to it. This is due to the same action that causes the expansion cracks shown in Figure 86, and can be considered identical in cause and effect.

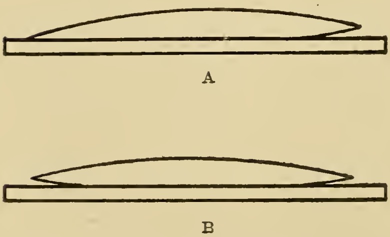


Fig. 87.—Pats Curling Away from the Glass.

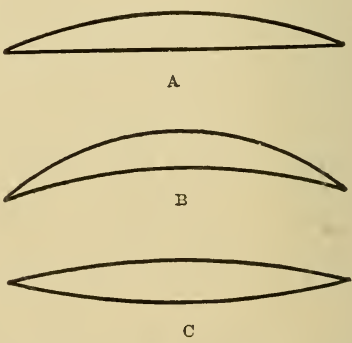


FIG. 88.—Pats Which Have Left the Glass, Showing Change in Volume.

Figure 88 shows pats which have left the glass (A) by lack of adhesion, (B) by contraction, and (C) by expansion. (A) is never dangerous in either air or water. (C) is only dangerous when existing in a marked form. (B) rarely, if ever, occurs in water, but in air often is indicatve of dangerous properties. Air pats developing this concave curvature generally disinte-

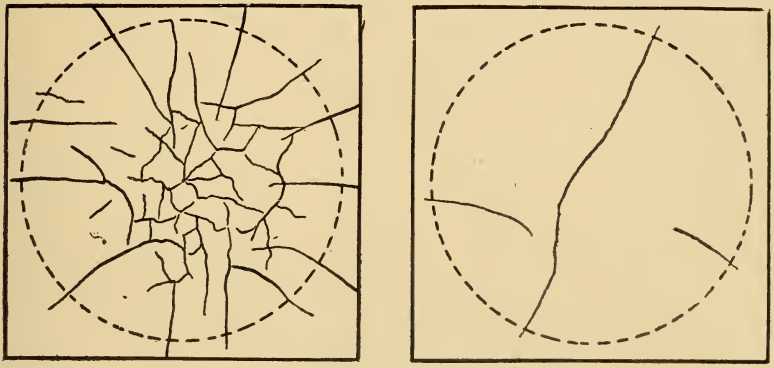


FIG. 89.—Pats Which Have Cracked the Glass.

grate eventually. A curvature of about a quarter of an inch can be considered about the limit of safety in a 3-inch pat.

Figure 89 indicates a peculiar condition in which the pat is perfectly sound and hard, but the glass on which it is made is badly cracked. This has been often erroneously attributed to chemical action, but is probably due entirely to the expansion of the pat, the adhesive strength of the cement to the glass exceeding the strength of the glass itself. It is only found in the water pats, and is rarely indicative of dangerous qualities.

Figure 90 represents blotching which usually is indicative of either underburning or adulteration. This condition should always be followed by an investigation of the causes producing it, which may or may not warrant rejection of the shipment.

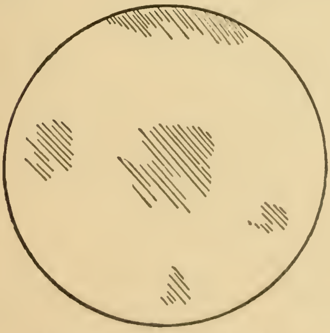


FIG. 90.—A Blotched Pat.

Figure 91 shows the radial cracks of incipient disintegration. These are the danger marks to be looked for in the normal pats, and their presence is always sufficient to warrant condemnation.

Figure 92 shows examples of complete disintegration, which almost invariably begins first by showing the radial cracks of Figure 91.

The great objection to the normal pats as an acceptance test, is the length of time often required for unsoundness to develop, cases being on record where disintegration only com-

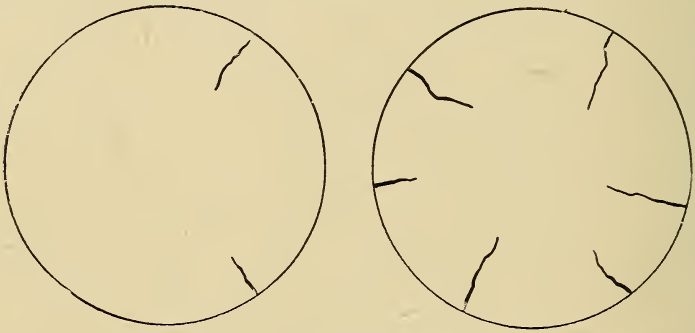


FIG. 91.—Radial Cracks.

menced after five or six years. In the author's laboratory, but 45 per cent. of the failures in the normal pats occur within 28 days. It is frequently necessary in practice to use a shipment of cement within a week after reception so that only a seven-day test is possible, and in such a case it is evident that the normal tests are almost worthless. It is to overcome this difficulty that the accelerated tests have been devised.

Accelerated Tests.—These accelerated tests are designed to hasten the action of the expansive ingredients and to produce the same results within a few hours or at most a week, for which

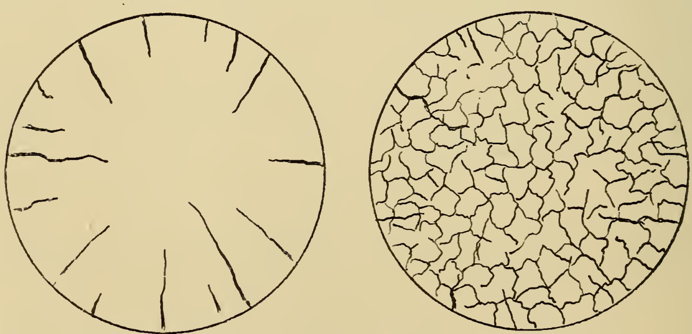


FIG. 92.—Disintegrated Pats (See also Figs. 99 and 100).

the normal pats require weeks, months or even years. Of the many varieties of accelerated tests the following are the best known and most used.

The Warm Water Test.—This was one of the first accelerated tests devised, and was proposed by Henry Faija in 1882. It consists in placing a pat, made of neat cement of normal consistency, and moulded on glass, in a closed vessel over water maintained at a temperature of 115 degrees Fahr., until it becomes hard set, after which it is lowered into the water for the remainder of 24 hours. The original apparatus designed by Faija is shown in Fig. 93, its construction being obvious.

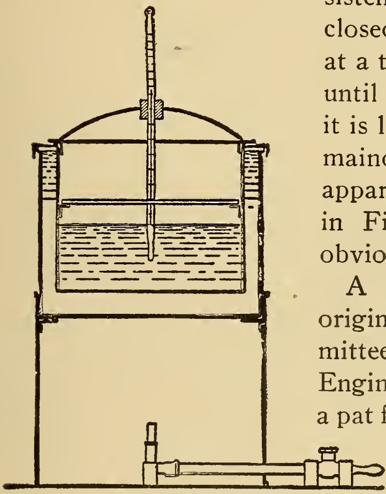


FIG. 93.—Faija's Hot Water Apparatus.

A modification of this test was originally recommended by the Committee of the American Society of Civil Engineers*, and consisted in keeping a pat for 24 hours in moist air, and then immersing it in cold water which was slowly raised to 115 degrees Fahr., and maintained there for 24 hours.

The Hot Water Test.—Since the temperature of 115 degrees Fahr. was often considered too low, Deval and others have advocated the same test, except for maintaining the water at a higher temperature, different experimenters recommending temperatures of all the way from 130 to 200 degrees Fahr.

Maclay's Tests.—These were the first accelerated tests used in the United States as a specification requirement, and consisted of making on glass plates six pats from neat cement of normal consistency. One of these as soon as made was placed in a steam vapor bath having a temperature of from 195 to 200 degrees Fahr.; the second put in the same bath as soon as it could bear the one-pound Gillmore needle; the third in double this time; the fourth after 24 hours. The fifth was placed in fresh water at 60 degrees Fahr. as soon as set, and the sixth kept in moist air at the same temperature. The first four of these pats remained in the vapor for three hours, then were immersed in hot water (about 200 degrees Fahr.) for 21 hours each, when they were taken out and examined. Mr. Maclay says "the

*This test was abandoned in the amendments adopted in 1904.

cracking or swelling of the first pat alone can generally be disregarded."

The Boiling Test.—This method was first proposed by Michaelis in 1870, and consists of making from neat cement paste, small balls about 5 centimeters in diameter. These balls are kept in moist air for 24 hours, then placed in cold water, which is gradually (in about half an hour) raised to boiling and maintained for six hours, when the specimens are removed and examined. Various experimenters have proposed many modifications of this test, some using pats instead of balls, others allowing the specimen more or less time to harden before testing, and still others making the duration of the test longer or shorter.

The Steam Test.—This test is recommended by the Committee of the American Society of Civil Engineers,* and consists of making a pat of neat cement paste of normal consistency, keeping it in moist air for 24 hours, then "exposing it in any convenient way in an atmosphere of steam, above boiling water in a loosely closed vessel, for 3 hours."

The Steam and Boiling Test.—A combination of the two foregoing methods is employed in several laboratories, consisting of putting a 24 hour old specimen in steam for 3 hours, then in boiling water for from 2 to 6 hours.

Other forms of accelerated tests less extensively used are the following:

The Kiln Test.—A cake of neat cement is made on a sheet of moist blotting paper upon a glass plate, and after it has set the cake is detached from the paper and preserved in moist air for 24 hours. It is then heated to 212 degrees Fahr. upon a metal plate in an air bath, heated with boiling water, until no more moisture is evolved, this requiring about 3 hours. This test has been modified by using a moist atmosphere in place of dry air.

The Heintz Ball Test.—A ball of neat cement paste about 2 inches in diameter is made and, when hard set, is placed on a thin iron plate above a Bunsen burner. The heat at first is gradually applied, then increased until the plate is red hot. The test is completed when no further moisture is evolved from the ball, this point being observed by condensation on a glass plate.

*See Appendix A.

Prussing's Press Cake Test.—This consists of making a dry mixture of neat cement, using from 5 to 8 per cent. of water, placing it in a mould and pressing it with a die under a load of 50 atmospheres. Two of these cakes are made, allowed to harden 24 hours in moist air, then one of them is used for the normal water test, and the other is placed in cold water for 2 hours, and then put into a water bath having a temperature of from 90 to 100 degrees C., where it is examined at the expiration of 4, and of 24 hours.

The Steam Pressure Test.—Erdmenger devised this method, which consists of allowing pats to harden 3 days in water at ordinary temperatures, then exposing them for 6 hours to steam at a pressure of from 3 to 20 atmospheres.

Deval's Hot Test.—This test consists of comparing the tensile strength of briquettes of sand mortar, one to three, at the age of 7 and 28 days when preserved in water at 15 degrees C., and at the age of 3 and 7 days when in hot water at 30 degrees C. It has also been proposed to test neat briquettes in a similar manner.

Le Chatelier's Test.—This method is to determine the expansion of a cylinder of cement which has been subjected to boiling.*

Chloride of Calcium Test.—Pats of cement are made of cement mixed with water containing 40 grammes of chloride of calcium to the liter, and, after setting, are immersed in the same solution for 24 hours.

In addition to the foregoing, many other methods for conducting accelerated tests have been devised, but their employment is too infrequent to warrant consideration.

Methods of Conducting Accelerated Tests.—With the possible exception of the Deval hot briquette test, only those forms of accelerated test made in steam or in hot or boiling water are employed in the United States in routine testing, and therefore the others will not be discussed further than to state that the results obtained from them give no better indication of good quality, and that in general they are more difficult to make and less easy to interpret properly.

The objection most frequently made to accelerated tests, and

*Described on page 161.

particularly to the boiling test, is the great variance in the methods used, different laboratories using different forms of test piece, and different periods of time before the test and for its duration. The variance in the results obtained under these several methods is, however, far less than it is generally supposed. The form of the test piece has practically little or no influence on the results, whether it be a cake, a ball, a wedge, or a pat, if the duration of the test be sufficient. The only advantage that the pat has over the other forms is that curvature can readily be ascertained, but a mere curvature of the pat should not be considered as a failure in boiling unless accompanied by checking. The requirement sometimes given that a pat should not leave the glass in boiling is not reasonable, as a small amount of expansion may naturally be considered normal.

The greatest difference in the methods of conducting the boiling test probably lies in the duration of the treatment, different specifications requiring the test to be made from one to as high as forty-eight hours. To determine the effect of different lengths of treatment a large number of tests on different cements were made by the author and the time at which failure occurred was observed. It was found that of those samples which did not pass the test, 22 per cent. failed in the first half hour, 57 per cent. failed in the first hour, 85 per cent. failed in two hours, 96 per cent. in three hours, and 99 per cent. in four hours. Only 1 per cent. of the tests that failed developed this action in over four hours, although many of them were carried up to twenty-four, and a few to forty-eight hours; thus showing generally that a test piece of cement standing three or four hours of boiling will almost invariably stand a much greater length of time, and also that at least three or four hours should always be allowed for the test.

The time allowed for the specimen to harden before it is tested may cause considerable differences in the results, but if it always be given time to fully develop hard set the differences will be slight. Pats of cement allowed more than about twelve hours to harden will, if unsound, fail when tested by boiling at almost any time in the future. The author has had normal pats as old as six months and apparently perfectly sound, which when put through the boiling test showed a failure almost identical with that obtained on the original test six months previously.

If, however, the specimen is tested before it has fully hardened, the differences obtained in the results are often very decided, and, curiously enough, may operate in either direction—that is to say, a pat of cement may fail more readily when one hour old than when twenty-four hours old, or a one-hour pat may pass the test, while the twenty-four-hour pat may fail. The reason for this action is by no means apparent, but it may be observed that, in the ordinary case of a cement high in free lime from underburning, the failure will usually be more marked in the fresher specimens, and that in the more infrequent case of a cement normally burned, but high in lime by reason of poor proportioning, the failure is often more marked in the older specimens. It would seem in this case that the cement was sufficiently strong to retain coherence in the test although insufficiently hardened, and that in this condition the lime was capable of becoming hydrated without causing disintegration.

For the same reasons a treatment of the specimen in a bath of steam before immersion in boiling water is generally less severe than if the specimen be boiled without this treatment, particularly so if the test be made before the test piece has become fully hardened.

It is also evident from the foregoing that tests made in steam alone without subsequent immersion in hot or boiling water may often give rise to erroneous conclusions regarding the results, especially if the specimens be tested soon after making. Tests made at temperatures below 140° or 150° Fahr., also, are not sufficiently severe to serve as an indication of quality, there being frequent cases on record in which samples have withstood the 115° hot water test and yet have failed in the normal pats in less than 28 days.

Apparatus.—Figure 94 shows an elaborate form of apparatus used in the Philadelphia Laboratories for conducting all hot water and steam tests. It consists of a double copper box covered with felt and asbestos. The inner tank contains two tiers of shelves of wire netting and is filled with water to a point between these tiers so that the test specimens may be either immersed or kept in the vapor above the water, which is maintained at a fixed height by means of constant level bottles. The space between the two boxes serves as a steam jacket into which the steam is introduced after passing through a pressure regulator. The temperature is controlled by a Heintz steam thermo-regulator so that the water may be kept at any fixed temperature for an indefinite time. For all but the largest laboratories, however, such an apparatus is unnecessarily elaborate.

Figure 95 shows a much simpler form of hot water bath, consisting of a double copper box, 18 ins. × 22 ins. × 18 ins., outside dimensions, and operating in manner exactly similar to that given before. The jacket, however, contains water, instead of

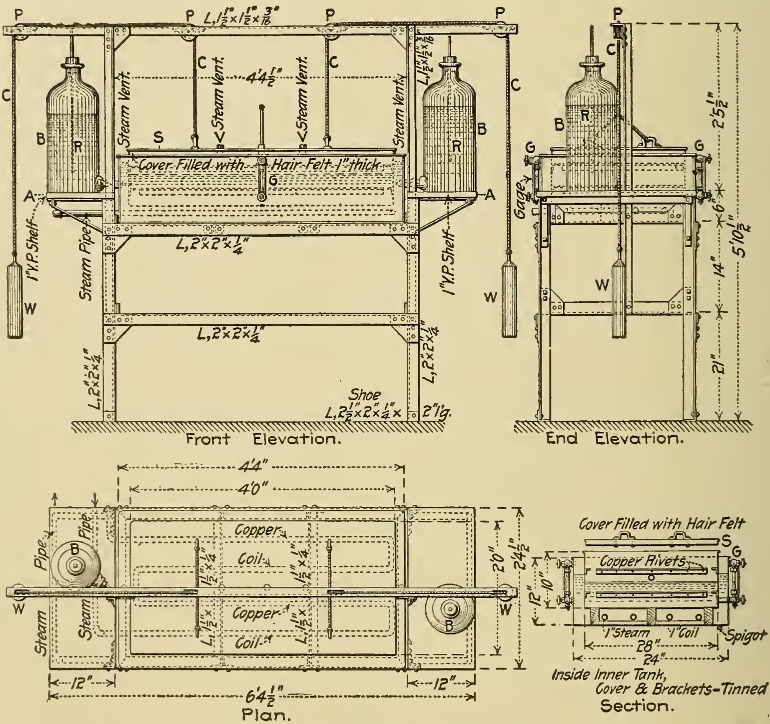


FIG. 94.—Apparatus for Accelerated Tests Used in the Philadelphia Laboratories.

steam, the heat being furnished by one or more Bunsen burners, the gas for which comes through a regulator, such as is shown in Fig. 96, and which is inserted in the vent at the top of the jacket. Excellent results may be obtained with this apparatus.

For boiling tests the simple copper box (6 ins. × 10 ins. × 7 ins.), shown in Fig. 97, is all that is necessary. A Bunsen burner furnishes the heat, and the screen of wire netting an inch above the bottom prevents the specimens from coming in contact with it. For steam tests, an exactly similar box is

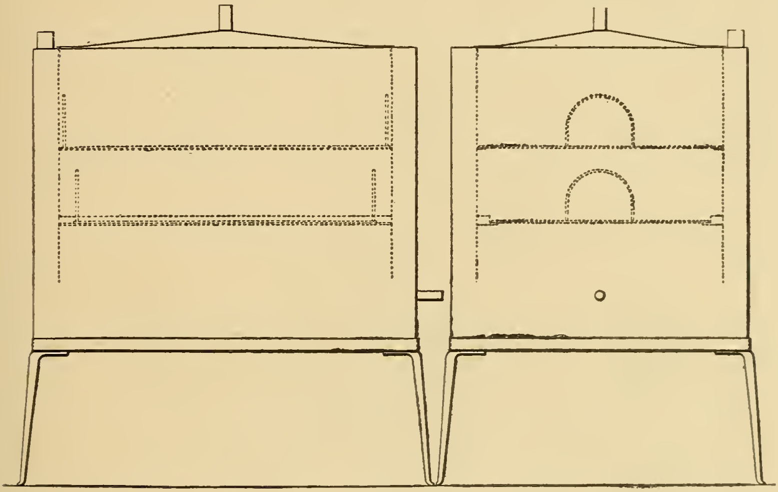


Fig. 95.—Simple Apparatus for Hot Water Tests.

used, except that the wire shelf is raised to a height of two inches above the water level. Any rough vessel may be used for occasional boiling tests, provided care is taken that the specimen does not come in contact with the bottom, and also that the evaporated water be replaced slowly, thus preventing a sudden chilling. Any permanently mounted apparatus for any of these tests should be provided with a constant level bottle as shown in Fig. 98, which is a large bottle provided with an opening at the bottom, and having at the top a tightly fitting rubber cork through which passes a glass tube. If the bottom of the bottle be connected with the boiling apparatus, the water will be maintained at a level equal to the height of the bottom of the glass rod.

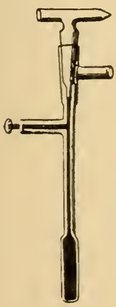


Fig. 96.—Gas Regulator.

Tests Used by the Author.—From all samples of Portland cement the author makes two pats 3½ inches in diameter, ⅜ inch thick at the center on plates of glass (4 ins. × 4 ins. × ⅛ in.), and also a small ball about 1¼ inches in diameter, which is shown in Figs. 80 and 81. These are made from neat cement paste of normal consistency, the material left over from the set test* being used for this purpose. They are marked as soon as made with a pointed piece of steel, and placed in the damp closet for 24 hours. At the expiration of that time, one of the pats is placed in water at a temperature of 65 to 70 degrees Fahr., and the other put in a closet protected from dampness, heat and sun's rays. The tanks used for storing the water pats are similar to those used for briquettes.† These two pats are examined

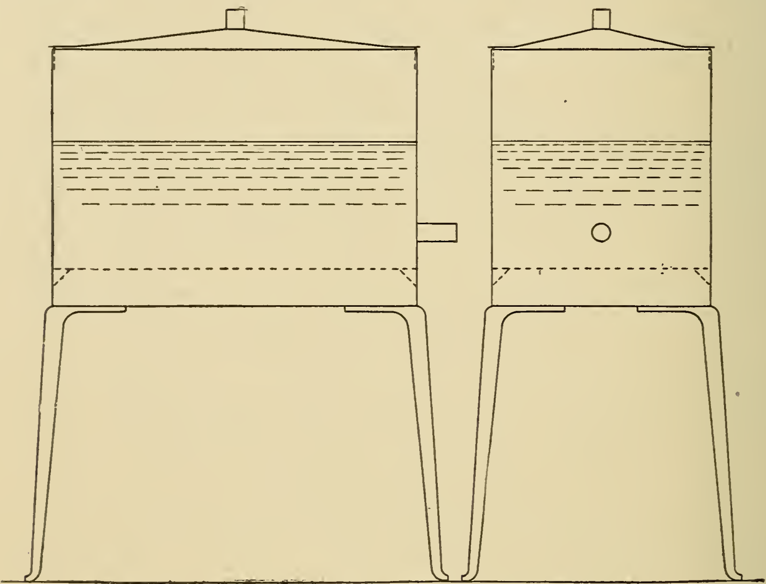


Fig. 97.—Apparatus for Making Boiling Tests.

at 7 days, 28 days, and thereafter at intervals of a month as long as they are kept, and their condition carefully recorded. The ball, as soon as it is removed from the damp closet, is placed in cold water in an apparatus similar to that shown in Fig. 97. The water is gradually (in about half an hour) raised to boiling, and maintained at that point for 3 hours, after which the specimens are removed and examined. Care must be taken to use fresh water every day, since, if repeatedly used, the water becomes strongly alkaline, sufficiently to often seriously affect the results.

*See page 98.
†See page 134.

Value of the Accelerated Tests.—Regarding the relation between the accelerated tests and the other tests for soundness and strength, there is but little question that the results are more or less corroborative. The author has recently compiled some data on this point covering over a thousand tests on many varieties of cement with the following results:

Of all samples failing to pass the boiling test, 34 per cent. of them developed checking or curvature in the normal pats or a loss of strength in less than twenty-eight days. Of those samples that failed in the boiling test, but remained sound at twenty-eight days, 3 per cent. of the normal pats showed checking or abnormal curvature in two months, 7 per cent. in three months, 10 per cent. in four months, 26 per cent in six months, and 48 per cent. in one year; and of these same samples, 37 per cent, showed a falling off in tensile strength in two months, 39 per cent. in three months, 52 per cent. in four months, 63 per cent. in six months, and 71 per cent. in one year. Or taking all these together, of all the samples that failed in the boiling test, 86 per cent. of them gave evidence in less than a year's time of possessing some injurious quality.

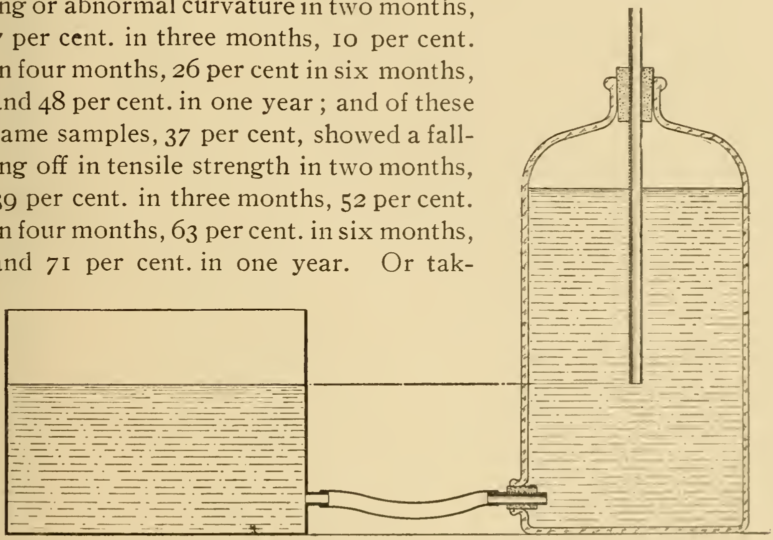


FIG. 98.—Illustrating the Principle of the Constant Level Bottle.

On the other hand, of those cements passing the boiling test, but one-half of 1 per cent. gave signs of failure in the normal pat tests, and but 13 per cent. showed a falling off in strength in a year's time.

To show roughly the relation in tensile strength of those cements failing and passing the boiling test, Table XLII. was compiled from 200 nearly consecutive tests of a single brand, 100 of them failing in the test and 100 passing. The high lime in those samples failing to boil is easily apparent in the high value of the seven-day neat test and its subsequent retrogression. While covering but a comparatively small number of tests, this table may, however, be considered fairly typical of the relations of strength to the accelerated tests, although exceptions, of course, frequently occur.

TABLE XLII.—Comparison of the Tensile Strength of Briquettes Failing and Passing in the Boiling Test.
(Tests by the Author.)

Age	Failing in Test		Passing Test	
	Neat	1 : 3 Sand	Neat	1 : 3 Sand
1 day	530	. .	391	. . .
7 days	817	197	643	237
28 "	749	273	727	303
2 months	713	274	732	312
3 "	702	242	749	314

In order to show the great value sometimes obtained from the results of the boiling test, several examples are given in Table XLIII. of tests of cements occurring in the regular routine work of the author's laboratory. The photographs of two of these tests are shown in Figs. 99 and 100. The first example is particularly remarkable in that at twenty-eight days there was absolutely no sign of failure whatsoever, except in the boiling test. All of these samples were normal in specific gravity, fineness, and time of setting, and both the tensile strength and the normal pats passed a good test at seven days, the boiling test giving the only indication of an unquestionable failure occurring at a later period. It should also be stated that these are not exceptional or "freak" cases, but examples of a common, although not frequent, occurrence.

Another point of considerable interest regarding the boiling test is this: The statement is often made that although a cement failing in this test may be otherwise sound, a cement passing the test may always be considered entirely safe, and while this is generally true, it is by no means an invariable rule. It occasionally happens that a cement may pass the boiling test well, and yet check and disintegrate in the normal tests, particularly if the cement be slow setting, high in lime, and the test made soon after the specimen is moulded. In these cases

TABLE XLIII.—Evidences of Failure in Cement Indicated by the Boiling Test. (Tests by the Author.)

Tensile Strength							Normal Pat Tests				Boiling Test
Neat				1·3 Sand			Air		Water		
1 day	7 days	28 days	4 months	7 days	28 days	4 mos.	28 days	4 months	28 days	4 months	
522	793	797	Disintegrated	204	257	52	Very slightly curled; left glass	Badly curled; soft and crumbly	Left glass	Disintegrated	Disintegrated
503	872	586	Disintegrated	184	239	47	Very slightly curled; left glass	Disintegrated	Left glass	Disintegrated	Disintegrated
498	762	700	Disintegrated	176	231	119	Very slightly curled; left glass	Badly curled; soft and crumbly	"O. K."	Disintegrated	Disintegrated
427	751	603	223	183	227	94	Very slightly curled; left glass	Disintegrated	"O. K."	Disintegrated	Disintegrated
503	827	717	177	220	252	132	Left glass	Badly curled; soft and crumbly	"O. K."	Disintegrated	Disintegrated
492	883	620	202	195	217	147	Left glass	Badly curled; soft and crumbly	"O. K."	Disintegrated	Badly checked
535	864	743	94	197	241	77	Very slightly curled; left glass	Badly curled; soft and crumbly	"O. K."	Badly curled and checked	Checked and cracked
502	829	722	320	203	247	65	Very slightly curled; left glass	Badly curled; soft and crumbly	"O. K."	Badly curled and checked	Checked and cracked
Neat tests not made				172	219	93	Very slightly curled; left glass	Badly curled; soft and crumbly	"O. K."	Badly curled and checked	Checked
Neat tests not made				198	231	101	Left glass	Badly curled; soft and crumbly	"O. K."	Badly curled and checked	Checked

Note.—All of these cements were normal in specific gravity, time of setting, and fineness.

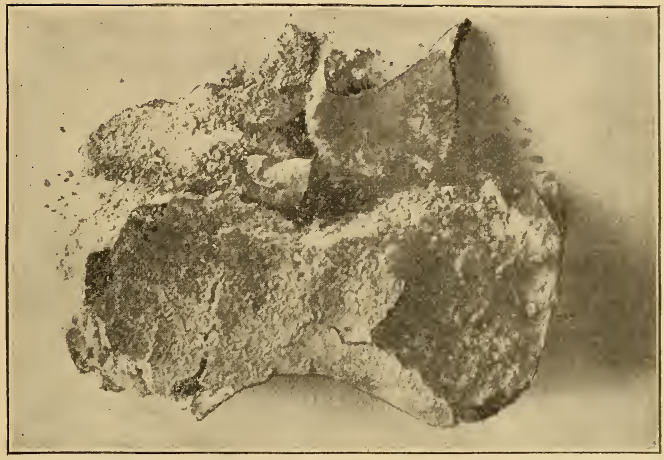
Briquette Kept in Water.

Normal Air Pat.

Normal Water Pat.

FIG 99.—Examples of Unsoundness Indicated by the Boiling Test. (See Table XLIII, No. 1.) Photographed at Four Months.

Briquette Kept in Water.



Normal Water Pat.

Normal Air Pat.

FIG. 100.—Examples of Unsoundness Indicated by the Boiling Test. (See Table XLIII, No. 2.) Photographed at Four Months.

it seems that the boiling at first tends to hydrate the lime and render it inert, although it would be active under normal conditions. It thus may be possible to add small quantities of lime to a sound cement and treat it in such a way that it will pass the boiling test perfectly, and yet fail under normal conditions. The author has seen the photograph of a test made in which as much as 15 per cent. of lime was added to a cement, and boiled, with excellent results, although the normal pats failed in a very short time.

Although it has been shown, however, that the results of the accelerated tests generally corroborate the other laboratory tests, it, nevertheless, cannot be denied that, in the vast majority of cases, work done with cement determined in the laboratory by means of the boiling test to be unsound will give

TABLE XLIV.—Examples of Retrogression in the Strength of Neat Briquettes, Without Similar Action in those of Sand Mortar.
(Tests by the Author.)

Neat					1 : 3 Sand			
1 day	7 days	28 days	4 mos.	1 year	7 days	28 days	4 mos.	1 year
450	782	705	392	203	180	252	309	314
493	717	801	317	241	172	247	283	297
429	685	793	593	318	209	251	298	323
473	791	790	502	291	192	267	287	301
502	823	752	421	200	184	229	252	251
423	802	741	511	277	212	237	290	303
479	784	782	520	321	202	238	301	318
461	791	797	493	290	178	251	277	276

most excellent results in practice, and show not the remotest sign of any sort of failure.

One reason that most cement shows such a radical difference in the results of the laboratory and in actual use is the fact that almost invariably the test is made considerably before the cement is used, a week almost always elapsing and often as much as a month, thus giving it plenty of time to season, and render the expansive elements ineffective, the shortest time customarily allowed, that of one week, being very often sufficient to make the difference between a radically unsound cement and one which is normal.

Another reason is that the disintegrating action of a cement is always far greater when mixed neat than when mixed with an aggregate, and the greater the amount of the aggregate the less the tendency to unsoundness. This can often be observed in the laboratory tests, cements often completely disintegrating in the neat briquettes, but retaining their strength in the sand tests. Table XLIV. shows a few instances of this sort. (See also Fig. 101.).

Even eliminating these two conditions, however, many cases are on record in which failure in boiling has not been corroborated by failure in the work, even though the cement was used at once and in a rich mixture, showing that even when the conditions of testing and actual work are most nearly alike, the indications of the accelerated tests are by no means infallible.

Concerning the value of the boiling test, the Report* of the Board of Engineer Officers (U. S. A.) says: "Of all these tests the boiling test is the simplest, requires only apparatus everywhere available, and is recommended by the Board. It has been the experience that this test detects material that is unsound by reason of the presence of active expansives; but in some cases it rejects material that would give satisfactory results in actual work and will reject material that would stand this test after air slaking."

"The great value of the test lies in its short-time indications and in at once directing attention to weak points in the cement to be further observed or guarded against. Of two or more cements offered for use or on hand, the cements that stand the boiling tests are to be taken preferably; it should be constantly applied on the work among other simple tests to be noted, for although the boiling test sometimes rejects suitable material, it is believed that it will always reject a material unsound by reason of the existence of active expansives. Sulphate of lime, while enabling cements to pass the boiling tests, introduces an element of danger."

"This test is proposed as suggestive or discriminative only. Except for works of unusual importance it is not recommended that a cement passing the other tests proposed shall be rejected on the boiling test."

A committee of the Society of German Portland Cement Manufacturers has reported: "After having made tests for the length of two years, the Commission Deciding as to the

*Professional papers, No.28, Corps of Engineers, U. S. A.

Constancy of Volume and the Adhesive Power of Portland Cement came to the conclusion that none of the so-called accelerated tests, boiling tests, etc., was capable of affording in

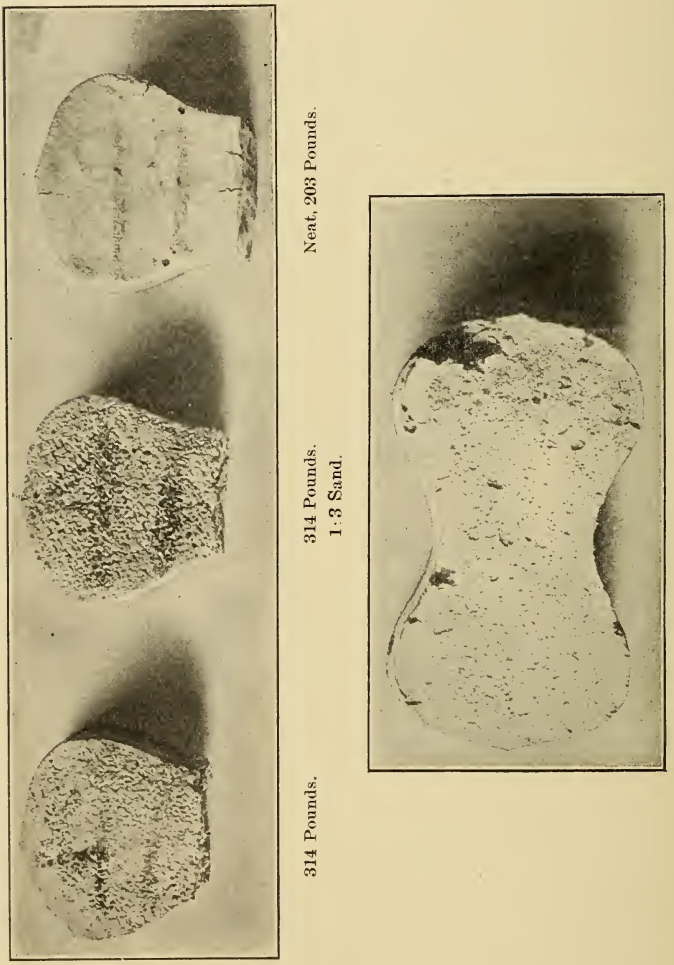


FIG. 101.—Neat Briquettes Disintegrating, while Sand Briquettes Remain Sound. (See Table XLIV, No. 1.) Photographed when One Year Old.

all cases a quick and reliable judgment in regard to the practical usefulness of a cement."

The Committee of the American Society of Civil Engineers* says: "In the present state of our knowledge it cannot be

*See Appendix A.

said that cement should necessarily be condemned simply for failure to pass the accelerated tests; nor can a cement be considered entirely satisfactory, simply because it has passed these tests."

Interpretation of Results.—To properly interpret the results of the soundness tests requires large and varied experience, and is undoubtedly the most difficult phase of the testing of cement.

Although not infallible, it is safe to consider the results of the normal tests, assuming correct manipulation, as absolute criteria of quality, and to reject all samples that fail to pass them.

If a sample fails in the accelerated test, as typified by the boiling test, it is the safe course to hold the shipment for at least 28 days, and then to make a second determination upon a fresh sample. If this second sample passes the test, it shows that the additional seasoning has made the shipment entirely sound and fit for use. If the second sample fails, and the neat tensile strength shows any decided retrogression in 28 days, the shipment should be considered as suspicious and probably unsafe, at least for the important parts of a structure. Generally, however, if all the other physical requirements are satisfied, and the boiling test alone fails, it is neither advantageous nor justifiable to reject the shipment, except, possibly, in a competitive test, in which case the samples passing the boiling test are to be considered preferable.

CHAPTER XI.

CHEMICAL ANALYSIS.

Components.—The components of Portland cement whose amounts are usually determined by chemical analysis are:—silica (SiO_2), alumina (Al_2O_3), iron (Fe_2O_3), lime (CaO), magnesia (MgO), and sulphuric acid (SO_3). Other ingredients less frequently determined are:—carbonic acid (CO_2), water (H_2O), alkalies ($Na_2O + K_2O$), and sulphur (S). In practice, alumina and iron are often determined together (written as R_2O_3), and carbonic acid and water together as "loss on ignition." The average amounts of these ingredients, together with a table of typical analyses have already been given in Chapter II.

Significance.—While chemical analysis plays a most important role in controlling the product in cement manufacture, it has comparatively small value in the testing of the finished material. This is due to the fact that the quality of the product depends not only upon the proportions of the different ingredients, but also upon their arrangement or the form of combination in which they exist. Thus it may happen that the content of lime, silica and alumina in a cement may be perfectly normal, and yet, by reason of defects in the process of manufacture, the material be of decidedly faulty character. For the detection of adulterants, or to determine whether certain constituents are present in amounts exceeding that believed to be safe, chemical analysis is of considerable value.

Analyses for silica, iron, alumina and lime are made in controlling the manufacture, but give little information in regard to the quality of the finished product, unless their proportions are grossly incorrect. Regarding this point the Committee of the American Society of Civil Engineers says:* "Faulty character of cement results more frequently from imperfect preparation of the raw material or defective burning, than from incorrect proportions of the constituents. Cement made from very finely ground material, and thoroughly burned, may contain much more lime than the amount usually present and still be perfectly sound. On the other hand, cements low in lime

*See Appendix A.

may, on account of careless preparation of the raw material, be of dangerous character. Further, the ash of the fuel used in burning may so greatly modify the composition of the product as largely to destroy the significance of the results of analysis."

Determinations of magnesia, sulphuric acid, sulphur, alkalies, and carbonic acid are made to ascertain whether these ingredients are present in inadmissible quantities. Best American practice in specifications limits magnesia to 4% and sulphuric acid to 1.75%.

In the following description of methods of chemical analysis, it is assumed that the reader has an elementary theoretical knowledge of chemistry, and is familiar with the ordinary processes of manipulation of chemical apparatus. No engineer, however well informed, should ever attempt chemical analysis without either a course of study in college, or a practical apprenticeship in some laboratory, for, unless he does so, it is impossible to obtain results of even approximate accuracy.

Methods of Analysis.—The general method given here for the analysis of Portland cement is in all essential particulars an elaboration of the method* proposed by the Committee on Uniformity in the Analysis of the Materials for the Portland Cement Industry of the New York Section of the Society for Chemical Industry, and which was indorsed† by the Committee on Uniform Tests of Cement of the American Society of Civil Engineers. This method is supposed to give the greatest accuracy consistent with a fair amount of rapidity, and, while scarcely practicable for control work on account of the many corrections, gives just about the degree of accuracy that should be obtained in an experimental laboratory. Following the system for general analysis are alternative methods that will enable the operator to secure greater refinement or greater rapidity as may be desired, and also short-cut methods suitable for control work.

GENERAL METHOD FOR THE ANALYSIS OF PORTLAND CEMENT, LIMESTONE AND RAW MATERIAL MIXTURES.

The sample should first be finely ground in an agate mortar, and a sufficient quantity for all determinations preserved in a tightly stoppered bottle.

*See Appendix B.
†See Appendix A.

Loss on Ignition.—0.5 gram of the sample is placed in a weighed platinum crucible and ignited, over a blast lamp, to constant weight. The crucible should be covered and the flame applied to the bottom at an angle of 45°. Ten minutes over a good blast should be sufficient for a cement and about 20 minutes for a limestone or slurry. The weight should be checked by 5 minutes further blasting.

Silica.—Having determined the loss, the ignited residue is transferred to a casserole and digested on a warm plate with 20 c. c. hydrochloric acid (1 — 1), until completely dissolved, the casserole being covered with a watch glass. The ignited cement or slurry should be entirely soluble in dilute acid; the presence of any gritty particles which can be felt with a stirring rod is an indication of incomplete solution. In such cases, these particles should be filtered off, the filter and contents being ignited in a platinum crucible. The residue should then be fused with a small quantity of sodium carbonate, the fusion taken up with hot water and added to the main solution, which is then evaporated to hard dryness. The residue is digested, for about 5 minutes, with 15 c. c. hydrochloric acid, after which the solution is made up to 100 c. c. with hot water. The separated silica is filtered off on an ashless filter and well washed with hot water, the filtrate and washings being caught in a second casserole. The filtrate is again taken to dryness, the residue treated exactly as before, and the small second precipitate of silica filtered off on a second filter. The two precipitates of silica are burned together in a weighed platinum crucible, first over the Bunsen burner until the carbonaceous matter is destroyed, and then over the blast for 15 minutes, after which it is cooled and weighed. Its weight should be checked by 10 minutes further blasting.

Silica almost invariably carries with it very small percentages of other constituents—usually iron and alumina. But as iron and alumina commonly contain small percentages of silica, which have not been removed by evaporation with hydrochloric acid, the errors practically counterbalance one another and, for ordinary purposes, a correction is not necessary. One constituent, moreover, should not be corrected without also correcting the others.

To determine the amount of impurity in the silica, it is moistened, after weighing, with a couple of drops of sulphuric acid, after which the crucible is filled about $\frac{1}{3}$ full with hydrofluoric acid and left on the hot plate until the contents have evaporated to dryness. The crucible is then carefully heated to redness, after which it is blasted for a few minutes, cooled and weighed. The loss in weight is pure silica—(SiO_2).

The residue is added to the iron and alumina. It is advisable to use the crucible containing the residue from the silica correction for the ignition of the iron and alumina precipitate.

Iron and Alumina.—To the filtrate from the silica, which should be made up to about 250 c. c., a very slight excess of ammonia is added; the solution is boiled to expel the excess of ammonia, after which the precipitate is allowed to settle. The solution should be filtered, while hot, into a large beaker, and the precipitate washed a couple of times with hot water. The funnel containing the iron and alumina is inverted and the precipitate carefully washed back into the original beaker with a spray of hot water. It is dissolved in hydrochloric acid, the solution diluted to 250 c. c. and the iron and alumina again precipitated with ammonia. The precipitate is then brought on the same filter and is thoroughly washed as before, the filtrate and washing being united with the first filtrate. In washing the gelatinous precipitate, the stream from the wash bottle should be so applied as to completely break up the mass each time and wash it free from the paper.

The precipitate and filter are transferred to a weighed platinum crucible, carefully ignited over a Bunsen flame until all the paper is burned off, after which it should be blasted for 5 minutes, cooled and weighed. The weighed residue is iron and alumina ($Fe_2O_3 + Al_2O_3$).

Iron Oxide.—The crucible containing the weighed oxides of iron and alumina is half filled with potassium bisulphate, covered and placed over a low Bunsen flame which is gradually raised as fusion proceeds. When fusion is complete and no more dark particles are floating about, the crucible is removed from the flame, manipulated so that the fusion runs up the sides, and cooled. When cold, the fused mass is transferred to a casserole, any adhering particles being washed out with hot water; 50 c. c. should be sufficient for solution of the fusion. Five c. c. concentrated sulphuric acid are added, and the solution is evaporated until fumes of SO_3 are evolved. It is allowed to cool, diluted with water to 50 c. c. and filtered from the separated silica, which is ignited, weighed, and its weight added to the original silica.

The solution, made up to 150 c. c., is placed in a flask and reduced, while hot, by hydrogen sulphide. The flask is then connected with a carbonic acid generator, and the excess of hydrogen sulphide boiled off in an atmosphere of carbon dioxide. Any separated sulphides are filtered off, after which the iron may be determined by titration with standard potassium permanganate.

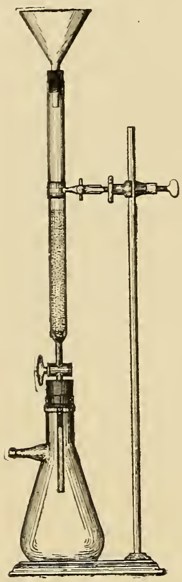


FIG. 102.—The Jones Reductor.

Some operators prefer reducing the solution with zinc; but in this case titanium is also reduced and determined with the iron, a difficulty which is obviated by reduction with hydrogen sulphide. The titanium may be determined colorimetrically after the titration.*

In making the reduction with zinc, the Jones reductor (Fig. 102) is the most convenient form of apparatus. The tube, having a small plug of mineral wool at the bottom, is filled to within a couple of inches of the top with pure shot zinc. To start the apparatus, the main tube containing the zinc is filled with 5% sulphuric acid, with the stop-cock open. When a good reaction has started, the solution of the bisulphate fusion is poured into the funnel, which should then be covered with a watch glass. Suction is applied at once to the flask, and the tube is kept filled, if necessary, by occasionally loosening the stopper at the top of the tube. The solution is never allowed to fall below the top of the zinc, and the level should be kept up by continual additions of water, being careful to rinse off the watch glass and the vessel which contained the solution. When all reaction in the tube ceases, the suction flask is disconnected, brought to the burette containing standard permanganate and the iron titrated.

Before beginning any determinations, blanks of acid and

*W. F. Hillebrand, Bulletin No. 176, U. S. Geological Survey.

water should be run through the reductor exactly as in making a determination, and titrated with permanganate until the amount required is found to be constant—about 0.1 to 0.2 c. c. This must be deducted from the amount required in analysis.

The permanganate solution may be standardized against iron wire, and should be made at least 24 hours before standardizing and kept in dark bottles. Two grams per liter is a good strength.

For standardizing, 0.2 gram of piano wire, having been polished with emery cloth and carefully cleaned, is made into a small coil and accurately weighed. In a 4 oz. Erlenmeyer flask provided with a valve stopper, 40 c. c. of 1—3 sulphuric acid are heated to boiling; the coil of wire is then dropped in, and the stopper quickly inserted with the valve open. When the wire has completely dissolved the solution is boiled for a couple of minutes, after which it is allowed to cool with the valve closed. When cold, the solution is transferred to a beaker, and the flask washed out a couple of times with cold water. Permanganate is then run into the solution from a burette until the pink color appears.

The weight of wire taken (less 0.3% for impurities) divided by the number of c. c. of permanganate equals the strength of the solution per c. c. in terms of iron. This result divided by 0.7 equals its strength in terms of Fe_2O_3.

Lime.—The combined filtrate and washings from the iron and alumina—about 500 c. c.—are brought to a *strong* boil on a hot plate, and, while boiling, there are added 25 c. c. of a hot saturated solution of ammonium oxalate. Boiling is continued for 5 minutes, after which the beaker is set aside in a warm place. When the precipitate has completely settled—which should be in about 20 minutes—the solution is filtered into a large beaker, the precipitate being brought on the filter paper and washed a couple of times with hot water. The funnel is then inverted and the precipitate washed back into the original beaker. The precipitate is dissolved in hydrochloric acid, and the solution diluted to 300 c. c. Five c. c. ammonium oxalate are added, the solution brought to a strong boil, after which ammonia is carefully added until the solution smells strongly of it. After boiling for 5 minutes, the beaker is set aside, and, when the precipitate has completely settled, it is collected on the same filter as before, washed several times with hot water, the filtrate and washings being united with those from the first precipitation.

The precipitate is dried by placing the funnel in an oven or supporting it over a hot plate. When dry, it is carefully brushed on a piece of black glazed paper, and the filter paper ignited in a weighed platinum crucible. When all the carbonaceous matter has burned off, the remainder of the precipitate is transferred to the crucible and the whole ignited to constant weight over a strong blast. With the crucible covered, twenty minutes over a good blast are usually sufficient. The crucible should be weighed as soon as cold. The final weight gives lime—CaO.

In precipitating the calcium oxalate, the solution should be *strongly boiling* before any attempt at precipitation is made, otherwise there will not be sufficient ebullition to keep the precipitate in suspension and the beaker will bump and likely break.

Magnesia.—The combined filtrate from the lime precipitation, after boiling for a few minutes to make sure that no more calcium oxalate comes down, is acidified with hydrochloric acid and evaporated on a hot plate to about 200 c. c. when 20 c. c. of a freshly prepared solution of microcosmic salt are added and the solution boiled for about 5 minutes longer. It is then transferred to a smaller beaker of suitable size and cooled by placing the beaker in a tray of ice water. When perfectly cool, ammonia is added a few drops at a time, with constant stirring, until the precipitate of magnesium ammonium phosphate is formed. A slight excess of ammonia is then added and the beaker set aside over night. The precipitate is filtered off and thoroughly washed with a solution of 1—4 ammonia. It is dried and ignited in a platinum crucible, first over a Bunsen burner, until most of the carbon is burned off, and then over a strong blast until perfectly white, being well broken up with a platinum rod. The ignited residue is magnesium pyrophosphate—$Mg_2P_2O_7$. Its weight multiplied by .362 gives the weight of magnesia—MgO.

The $Mg_2P_2O_7$ should be completely soluble in hot hydrochloric acid, but is likely to contain amounts of silica dependent on the quality of the glassware used for evaporating. In this event, the silica is filtered off, the filtrate and washings caught in a small beaker and diluted to 100 c. c. Three c. c. of saturated microcosmic salt solution are added and the magnesium ammonium phosphate reprecipitated by the addition of ammonia and treated in the manner previously described.

Sulphuric Acid.—For this determination a separate sample of 0.7 gram is taken and digested on a warm plate with 50 c. c. of 1—4 hydrochloric acid until nothing remains in suspension other than a few particles of flocculent insoluble silicious matter. This is filtered off, the filtrate and washings made up to about 200 c. c. and brought to boiling. Ten c. c. of a hot 10% solution of barium chloride are then added, with brisk stirring, and the boiling continued for 5 minutes, after which the covered beaker is set aside for several hours on a warm plate—about 50° C. When the precipitate has completely settled, it is filtered on a small paper, washed several times with hot water and then carefully ignited, with the filter, in a weighed platinum crucible over a Bunsen flame until perfectly white. This weighed precipitate is barium sulphate—$BaSO_4$—multiplied by .343 gives the weight of sulphuric anhydride—SO_3.

Allowing the precipitate to settle out in a warm solution has been found advisable, since it is then easily retained by the filter; otherwise it is apt to run through and give considerable trouble.

ANALYSIS OF CEMENT ROCK, CLAY AND NATURAL CEMENT.

In cases where the substance is not decomposible by hydrochloric acid, recourse must be made to fusion with sodium carbonate. A 0.5 gram sample, after the loss on ignition has been determined, is intimately mixed with about 10 times its weight of sodium carbonate, with which a few small crystals of potassium nitrate have been ground. The mixture is transferred to a platinum crucible with a tight fitting cover and heated over a strong flame until it is in a state of quiet fusion. A few minutes over the blast lamp is advisable before cooling. The fusion is then run up the sides of the crucible, and allowed to cool, after which the crucible, with its contents and the lid, are placed in a casserole, covered with water and allowed to digest on a hot plate until the fusion is well disintegrated. The crucible and lid are then removed, washed, using hydrochloric acid if necessary, and sufficient hydrochloric acid is carefully added to bring everything into solution. The solution is then evaporated to dryness for the separation of silica, after which the procedure is exactly similar to that used for Portland cement and limestones. The precipitates, however, all require considerably more washing owing to the fixed alkali from the fusion which is difficult to wash out. The inexperienced operator should test a few drops of the washings with silver nitrate—a precipitate of silver chloride indicates that the washing has been insufficient.

ALTERNATIVE AND ADDITIONAL METHODS.

Lime—Volumetric Method.—In mill laboratories and where greater rapidity is necessary, the lime generally is determined volumetrically. The method depends on the following reaction:

$$5\ H_2C_2O_4 + 2\ KMnO_4 + 3\ H_2SO_4 = K_2SO_4 + 2\ MnSO_4 + 8\ H_2O + 10\ CO_2.$$

Six grams per liter is a convenient strength for the permanganate solution. It should be made not less than 24 hours—preferably several days—before standardizing, should frequently be well shaken during that period and kept in dark bottles. It may be standardized against oxalic acid. The acid, however, should be recrystallized, dried between filter paper and finally in a current of dry air, and preserved in a tightly stoppered bottle. 0.2 gram of the crystals so prepared is dissolved in 100 c. c. of water; 5 c. c. concentrated sulphuric acid are added and the solution brought just to a boil. The permanganate solution is then run in from a burette until the pink color is permanent. The weight of acid taken divided by the number of c. c. permanganate solution used gives the value of the solution in terms of oxalic acid. This result multiplied by 0.444 gives the value in terms of CaO. Duplicate determinations should not vary more than 0.00002.

When pure calcite ($CaCO_3$) can be obtained, it is preferable to dissolve a weighed quantity, precipitate the lime by ammonium oxalate, and standardize the solution against it as in the regular course of analysis.

For the determination of the lime, the precipitate of calcium oxalate is obtained as for the gravimetric method. It is washed five or six times with hot water, or until a few drops of the washings fail to decolorize a similar quantity of dilute permanganate. The funnel is then inverted and the precipitate washed back into the original beaker, after which the filter paper is spread out on the inner side of the beaker, and washed with 1—4 sulphuric acid to dissolve any adhering particles of precipitate, followed by hot water. The precipitate in the beaker is then stirred with a spray of the 1—4 sulphuric acid until solution takes place.

It is diluted to 200 c. c., brought barely to a boil, and titrated with standard permanganate. The number of c. c. taken multiplied by the CaO value gives the amount of lime.

Magnesia—Volumetric Method.—The simplest method depends on the following reaction:

$$Mg\,(NH_4)\,PO_4 + H_2SO_4 = (NH_4)\,H_2PO_4 + MgSO_4.$$

The filtrate from the lime precipitate having been considerably reduced in volume by evaporation, is transferred to a large Erlenmeyer flask and made strongly ammoniacal. 20 c. c. of a saturated solution of microcosmic salt are then added and the mixture is thoroughly shaken until the precipitate is well formed. The precipitate is allowed to settle, filtered out and washed as usual. The paper is then spread out on a bisque plate. This will rapidly absorb the greater part of the water and ammonia, after which the paper is removed from the plate and dried in an oven at 50° or 60° C., for fifteen minutes. When this is accomplished, the paper and precipitate are thrown into a beaker and an excess of $\frac{N}{10}$ sulphuric acid added. When the precipitate has entirely dissolved, the excess of acid is titrated back with $\frac{N}{10}$ ammonia.

$$1 \text{ c. c. } \frac{N}{10}\ H_2SO_4 = .002\ MgO.$$

Mr. R. K. Meade* has evolved a method, briefly described by Sutton† as follows: "When a solution of arsenic acid contains sufficient sulphuric or hydrochloric acid, the arsenic is quickly reduced to arsenious acid even in the cold. For every

*"Examination of Portland Cement," by R. K. Meade.
†"Volumetric Analysis," by Francis Sutton.

molecule of arsenic acid so reduced there corresponds two atoms of magnesium, two molecules or four atoms of iodine are liberated. This latter is titrated with sodium thiosulphate, and from the volume of standard solution required, the magnesium calculated.

"The standard solutions are conveniently made as follows:

"Standard sodium arsenate is prepared by dissolving 12.29 grams of pure arsenious acid in nitric acid, evaporating on a water-bath to dryness, neutralizing with sodium carbonate in solution, and when dissolved made up to a liter with distilled water. Each c. c. = 0.005 gm. of MgO.

"The standard solution of sodium thiosulphate is made to correspond to this either by direct titration, or by making it equal to a standard iodine solution made by dissolving 52.24 gm. of pure iodine, and 75 gm. of potassium iodide in about 200 c. c. of water, and making up to one liter. Each c. c. = 0.005 gm. MgO.

"Process.—Pour the magnesia solution, which should not contain too great an excess of ammonium chloride or oxalate into an Erlenmeyer flask or a gas bottle of sufficient size. Add one-third the volume of the solution of strong ammonia and 50 c. c. of sodium arsenate. Cork up tightly and shake vigorously for ten minutes. Allow the precipitate to settle somewhat, then filter and wash with a mixture of water and strong ammonia (3—1) until the washings cease to react for arsenic; avoid, however, using an excess of the washing fluid. Dissolve the precipitate in dilute hydrochloric acid (1—1), allowing the acid solution to run into the flask in which the precipitation was made, and wash the filter paper with the dilute acid, until the washings and solution measure 80 or 100 c. c. Cool, and add from 3 to 5 gm. of potassium iodide, free from iodate; allow the solution to stand a few minutes, and then run in the standard thiosulphate until the color of the liberated iodine fades to a pale straw color. Add starch, and titrate until the blue color of the iodide of starch is discharged. If preferred, an excess of thiosulphate may be added, then starch and standard iodine until the blue color is produced. On adding the iodide of potassium to the acid solution, a brown precipitate forms, which, however, dissolves when the thiosulphate is added.

"Experience has proved that the whole process can be done within an hour, and the results are very near those given by gravimetric methods."

Sulphuric Acid.—Mr. D. D. Jackson has devised* a photometric method for this constituent depending upon the turbidity of a solution holding barium sulphate in suspension. The cement is dissolved as in the regular method of determination, and the solution transferred to a 100 c. c. Nessler tube. When cold, crystals of barium chloride are added, after which the tubes are corked, well shaken, and set aside for a short time.

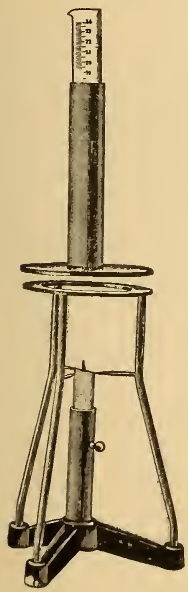


FIG. 103.—Jackson's Apparatus for Determining Sulphuric Acid.

For the determination there is a tube graduated into millimeters and inclosed in an opaque sleeve to shut out the light, Fig. 103. It is suspended in a rack so that the bottom is 3 inches above the flame of a standard candle; and on looking down through the tube there is visible a bright circle of light.

To make the determination, the precipitated solution in the Nessler jar is made up to the 100 c. c. mark and thoroughly agitated. Sufficient of it is then slowly poured into the graduated tube until the last addition just shuts out the circle of light at the bottom of the tube. This point is very well defined. For the number of millimeters of solution required, a table prepared by Mr. Jackson gives the corresponding amount of sulphuric acid.

While the method cannot be called an exact one, the results are accurate enough for ordinary purposes, and the method is convenient where a great number of determinations are required.

Total Sulphur.—Sulphur in clay and rock usually exists as iron pyrites, FeS_2. For its determination, 1 gram of the finely pulverized sample is intimately mixed with about ten times its weight of a mixture of 10 parts sodium carbonate and 1 part potassium nitrate. The mixture is transferred to a tightly covered platinum crucible and heated to quiet fusion over a strong Bunsen flame. It is advisable to have the crucible

*Journal American Chemical Society, Vol. XXIII., No. 2.

placed in a hole through an asbestos board, in order that the gas from the flame may not contaminate the fusion.

The fusion is treated exactly as for the complete analysis of cement rock, etc., excepting that when it has gone into solution upon the addition of hydrochloric acid, the solution is filtered into a beaker of suitable size, brought to a strong boil, and the sulphur (now oxidized to sulphate) precipitated, while

TABLE XLV.—For the Reduction of Observations Made With Jackson's Sulphate Apparatus.
(Compiled by Mr. Jackson.)

Depth Cm.	Per Cent. SO_3.	Depth Cm.	Per Cent. SO_3.	Depth Cm.	Per Cent. SO_3.	Depth Cm.	Per Cent. SO_3.
1.0	5.2	4.0	1.4	7.0	0.8	10.0	0.6
1.1	4.8	4.1	1.4	7.1	0.8	10.2	0.6
1.2	4.4	4.2	1.3	7.2	0.8	10.4	0.6
1.3	4.1	4.3	1.3	7.3	0.8	10.6	0.5
1.4	3.8	4.4	1.3	7.4	0.8	10.8	0.5
1.5	3.6	4.5	1.3	7.5	0.8	11.0	0.5
1.6	3.4	4.6	1.2	7.6	0.8	11.2	0.5
1.7	3.2	4.7	1.2	7.7	0.7	11.4	0.5
1.8	3.0	4.8	1.2	7.8	0.7	11.6	0.5
1.9	2.9	4.9	1.2	7.9	0.7	11.8	0.5
2.0	2.7	5.0	1.1	8.0	0.7	12.0	0.5
2.1	2.6	5.1	1.1	8.1	0.7	12.2	0.5
2.2	2.5	5.2	1.1	8.2	0.7	12.4	0.5
2.3	2.4	5.3	1.1	8.3	0.7	12.6	0.5
2.4	2.3	5.4	1.0	8.4	0.7	12.8	0.4
2.5	2.2	5.5	1.0	8.5	0.7	13.0	0.4
2.6	2.1	5.6	1.0	8.6	0.7	13.5	0.4
2.7	2.1	5.7	1.0	8.7	0.7	14.0	0.4
2.8	2.0	5.8	1.0	8.8	0.6	14.5	0.4
2.9	1.9	5.9	1.0	8.9	0.6	15.0	0.4
3.0	1.9	6.0	0.9	9.0	0.6	15.5	0.4
3.1	1.8	6.1	0.9	9.1	0.6	16.0	0.4
3.2	1.7	6.2	0.9	9.2	0.6	16.5	0.4
3.3	1.7	6.3	0.9	9.3	0.6	17.0	0.3
3.4	1.6	6.4	0.9	9.4	0.6	17.5	0.3
3.5	1.6	6.5	0.9	9.5	0.6	18.0	0.3
3.6	1.6	6.6	0.9	9.6	0.6	18.5	0.3
3.7	1.5	6.7	0.8	9.7	0.6	19.0	0.3
3.8	1.5	6.8	0.8	9.8	0.6	19.5	0.3
3.9	1.4	6.9	0.8	9.9	0.6	20.0	0.3

boiling, with 10 c. c. of 10% barium chloride solution. After boiling 5 minutes longer, the beaker is set aside in a warm place. When the precipitate has completely settled it is filtered off, ignited and weighed.

The weight multiplied by .258 gives the amount of iron pyrites FeS_2. However, any sulphate sulphur, determinable by the method of solution in hydrochloric acid, should have its corresponding weight of barium sulphate subtracted from the above weight before multiplying by the factor.

Sulphur (as Calcium Sulphide).—While this determination is not frequently made, it is sometimes desirable, especially with slag cements. Five grams of the sample are introduced into a 6-ounce Erlenmeyer flask, provided with a rubber stopper, having a small tap funnel and a delivery tube leading to the bottom of an 8-inch test tube. The test tube is $\frac{2}{3}$ filled with an ammoniacal solution of cadmium chloride; 50 c. c. of 1—1 hydrochloric acid are run into the Erlenmeyer flask through the tap funnel, the solution gradually heated to boiling and boiled for several minutes. The hydrogen sulphide evolved precipitates cadmium sulphide in the test tube. When the solution has boiled sufficiently, the delivery tube is disconnected, and the contents of the test tube transferred to a casserole. A drop of starch solution is added and sufficient dilute hydrochloric acid to dissolve the cadmium sulphide; $\frac{N}{10}$ iodine solution is then run in from a burette until a blue color appears.

$$1 \text{ c. c. } \frac{N}{10} \text{ iodine} = .0036 \text{ calcium sulphide } (CaS).$$

Alkalies.—For the determination of the alkalies the method of J. Lawrence Smith is generally employed, as follows:

One gram of the finely divided sample is intimately mixed with an equal weight of ammonium chloride. Eight grams of precipitated calcium carbonate are then thoroughly incorporated with the mixture; the mass is transferred to a capacious platinum crucible with a tight fitting lid, and heated over a Bunsen flame. The heat is applied gently at first, until fumes of ammonium salts cease, after which the crucible is heated to a bright red for one hour. After cooling, the fusion is transferred to a platinum dish, covered with water and allowed to slake. When thoroughly slaked, the solution is filtered into another dish, and evaporated to about 50 c. c., when 2 grams ammonium carbonate are added. As soon as the ebullition ceases, the clear liquid is filtered off into a weighed platinum dish, and evaporated. A crystal of ammonium carbonate should be added during the evaporation; if a precipitate separates it must be filtered. When there is no further precipitation, the solution is acidulated with hydrochloric acid and taken to dryness on the water bath. When perfectly dry, the chlorides are broken loose from the dish and carefully ignited at a dull red heat to constant weight. This gives the combined chlorides—NaCl + KCl.

For their separation, the mixed chlorides are taken up with a few c. c. of water and a drop or two of hydrochloric acid, and heated on the water bath. There are frequently a few grains of insoluble matter; this should be filtered off and ignited, and its weight deducted from that of the combined chlorides. To the solution of the chlorides, 1 to 2 c. c. of a solution of platinic chloride are added, after which it is evaporated on the water bath until crystallization begins. A few c. c. of water and an equal quantity of alcohol are added; the insoluble potassium platinic chloride is filtered off on a weighed Gooch crucible or tared filter, washed with alcohol until the washings are colorless, dried at 100° C. for one hour, and weighed as potassium platinic chloride (K_2PtCl_6). We then have:

$$\text{Wt. } K_2PtCl_6 \times .194 = K_2O.$$
$$\text{Wt. } K_2PtCl_6 \times .307 = KCl.$$
$$\text{Wt. } NaCl \times .531 = Na_2O.$$

Carbon Dioxide.—For the accurate determination of carbon dioxide in rocks or cements, a train, such as is represented in Fig. 104, is employed. It consists of a washing bottle, containing dilute sulphuric acid, followed by a tower of caustic potash connected to a tap funnel leading to the bottom of a 4-oz. Erlenmeyer flask. From this flask a tube leads to an upwardly inclined condenser, followed in this order by a "U" tube of calcium chloride, a tube of anhydrous copper sulphate, a second tube of calcium chloride, and then the two weighed "U" tubes, filled with soda lime for the absorption of the carbon dioxide and provided with stop-cocks. These are followed by a "U" tube containing soda lime in its inner and calcium chloride in its outer arm, which leads to a suction pump or aspirator bottle. Before using the apparatus a current of CO_2 should be passed through the first three "U" tubes, in order to saturate any free lime. It should be followed by a liter of purified air.

To operate the apparatus, a weighed portion of the sample, ranging from 0.5 gram of a limestone to 5 grams of Portland cement, is introduced into the Erlenmeyer flask and covered with water. All the stop-cocks are opened and a liter of air is aspirated through the apparatus. The soda lime tubes are then removed (with stop-cocks closed) and placed in the balance case for 15 minutes, after which they are weighed. The operation is repeated until both tubes reach a constant weight.

Fifty c. c. of 1—1 hydrochloric acid are then introduced into the tap funnel, and, with the train connected up, allowed to run into the flask. When the reaction becomes weak, air is slowly aspirated through, the solution in the flask gradually brought to boiling and allowed to boil for several minutes,

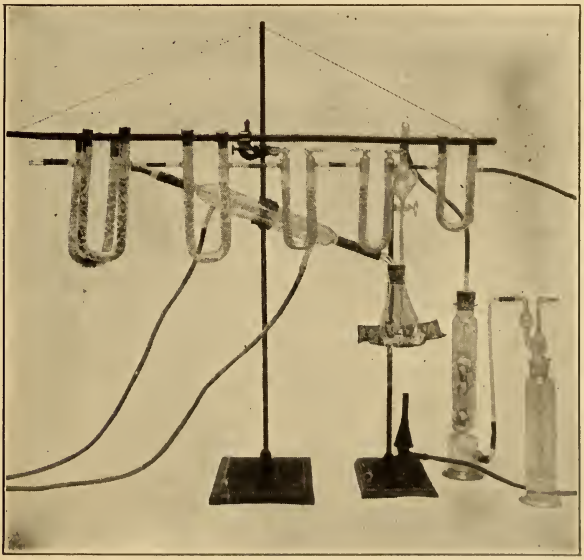


FIG. 104 —Apparatus for Determining Carbon Dioxide.

after which the flame is removed and a couple of liters of air aspirated. The stop-cocks are then closed, the weighed tubes removed to the balance case, and, after standing 15 minutes, are weighed. They are then again connected with the train, and, after a liter of air is aspirated, weighed, and this is repeated until a constant weight is attained. The gain in weight is carbon dioxide—CO_2.

For more rapid work, some operators prefer the small gas bottles, an excellent form of which is shown in Fig. 105. A weighed quantity of the sample is introduced into the lower part of the bottle and covered with water. The inlet tube which leads to the bottom of the flask is filled with 1—1 hydrochloric acid, while the other is $\frac{1}{3}$ filled with strong sulphuric acid to absorb any moisture that might escape during the operation. When everything is prepared the bottle is wiped clean and weighed, after which the hydrochloric acid is admitted to the sample. When the first reaction is over, a light suction is applied to the outlet and air is aspirated until the bottle reaches a constant weight. The loss from the original weight is carbon dioxide—CO_2.

Carbon Dioxide and Water.—For the determination of both these constituents in a cement or rock, the Shimer crucible (Fig.106) is found very convenient, and its operation is described at some length by Meade.* The newer form of crucible is a considerable improvement over the old type in that it is provided with a circulating chamber for water. The crucible is set up in a train having on one side an aspirator, a calcium chloride and a caustic potash jar to purify the air entering the apparatus, and on the other side a weighed calcium chloride tube followed by a weighed potash bulb and guard tube filled with calcium chloride.

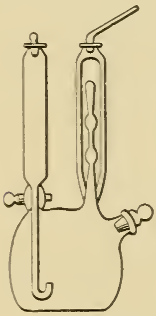


Fig. 105.—Apparatus for Determining Carbon Dioxide by Loss of Weight.

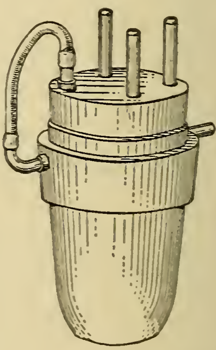


Fig. 106.—The Shimer Crucible.

The weighed sample is placed in the crucible, and the cap is made air tight by means of a rubber band between it and the crucible. A circulation of water keeps this band from becoming hot. With a slow current of air passing through the apparatus, the crucible is heated by a strong Bunsen burner for 10 minutes, followed by a blast for 20 minutes longer. The lamp is then removed, after which the aspiration is continued for about 10 minutes. The tubes are then disconnected and weighed, observing the same precautions as in the preceding methods.

*"Examination of Portland Cement," by R. K. Meade.

RAPID METHODS FOR CONTROL WORK.

In making complete analyses of the raw materials or the finished product, the methods customarily followed in the mills are practically a condensation or simplification of the preceding general method. Corrections are never applied, the silica is evaporated but once and generally baked for 15 or 20 minutes; only one precipitation is made for lime and magnesia, the former of which is determined volumetrically, while both gravimetric and volumetric methods are employed for magnesia. The accuracy thus obtained is less than that of the general method, but the errors are usually systematic and hence are comparatively unimportant in control work which requires the determination of variations, rather than absolute quantities.

For the proportioning of the raw materials, the lime alone is customarily determined, it being assumed that the other ingredients vary in more or less of a fixed ratio. Proportioning is occasionally fixed by the silica content, but the use of this method is infrequent, and much less accurate and satisfactory. For the rapid determination of lime, the following two methods* represent the best practice, the first method depending upon titration with potassium permanganate, and the second upon titration with standard acid and alkali solutions.

"**Method 1.**—In American practice this method is used most commonly and seems to enjoy the greatest favor among cement chemists. It requires a potassium permanganate solution of such strength that 1 c. c. = 0.005 gram calcium carbonate or calcium oxide, depending on whether a raw mixture or burnt cement is to be analyzed. The method is carried out for limestone as follows:

"Weigh out 0.5 gram of the finely ground sample into a platinum crucible and ignite over the Bunsen burner to destroy all organic matter. Transfer the sample to a 300 c. c. beaker, add 30 c. c. of water, cover with a watch glass, add 10 c. c. of hydrochloric acid and a little nitric acid. Boil till all the soluble matter is dissolved and all the carbon dioxide expelled. Wash off watch glass and dilute to about 150 c. c. with water previously boiled. Add ammonia slightly in excess and heat to boiling. If the insoluble residue is low and it is not desired

*From "The Manufacture of Portland Cement," by A. V. Bleininger.

to weigh the insoluble matter it is not necessary to filter it off. The calcium oxalate is precipitated in the boiling hot solution as usual by the addition of 40 c. c. of a hot solution consisting of 20 c. c. of concentrated ammonium oxalate solution and 20 c. c. of water. Stir for several minutes and let settle for five minutes.

"Decant the supernatant solution through an ashless filter, add 40 c. c. of hot water, decant, add another portion of hot water and decant for the third time. Now transfer the precipitate to the filter and wash three or four times with hot water. To determine whether or not the precipitate has been washed sufficiently, catch a few c. c. of the last filtrate on a watch glass, add a drop of sulphuric acid and one drop of potassium permanganate solution. If the liquid shows a strong red color the washing is finished, if the color is discharged further washing is necessary. The calcium oxalate is now washed back into the beaker in which it was precipitated, using hot water and diluted to about 200 c. c., if necessary. Place the beaker under the funnel and run through the filter into the beaker 30 c. c. of dilute sulphuric acid (1 volume of acid to 3 of water). Wash the filter thoroughly with hot water and stir the contents of the beaker while running in the acid. Heat liquid to about 80° degrees C. and titrate with the permanganate solution to a faint pink color which should not disappear for two minutes.

"The potassium permanganate solution should not be standardized against iron or ammonium ferrous sulphate, but against calcite checked repeatedly by the gravimetric method of calcium determination.

"R. K. Meade* proposes to keep the iron and alumina in solution by the addition of 5 per cent. oxalic acid, the calcium being precipitated by ammonium oxalate and determined volumetrically with a standard permanganate solution. The results have been found by Meade to be very satisfactory.

"**Method 2.**—The acid alkali methods, owing to their rapidity and simplicity, are frequently made use of, but great caution is necessary in their use, and the results should be carefully checked gravimetrically from time to time owing to the fact that these methods are subject to errors. Larger amounts of

*"Cement and Engineering News," June, 1903.

alumina and iron influence the results most decidedly. S. B. Newberry* proposes the following working method:

"Prepare a n/5 solution of hydrochloric acid and a n/5 caustic soda solution, standardizing with pure Iceland spar, which has been analyzed gravimetrically. One-half gram of pure spar should exactly neutralize 50 c. c. of acid.

"Weigh out $\frac{1}{2}$ gram of a finely ground limestone, transfer to an Erlenmeyer flask of about 500 c. c. capacity provided with a rubber stopper and a thin glass tube 30 inches long, to serve as a condenser. Run into the flask 60 c. c. of the 1-5 normal acid, attach the condenser and boil gently, allowing no steam to escape from tube, for about two minutes. Wash down the tube into the flask with a little water. Remove the condenser and cool the solution thoroughly by immersing the flask in cold water. When quite cold add five to six drops of phenolphthalein solution (1 gram in 200 c. c. alcohol) and titrate back to first pink color with 1-5 normal caustic soda solution. It is important to recognize the point at which the first pink color appears throughout the solution, even though this may fade in a few seconds. If the alkali be added to a permanent and strong red color the lime will come too low. The amount of acid used is called the first acid and the alkali used to titrate back, the first alkali.

"In case the materials contain a very small amount of magnesia the operation ends here and the calculation is simply: Number of c. c. acid minus number of c. c. alkali multiplied by $2 \times 0.56 =$ per cent. calcium oxide. In this case it is unnecessary to cool the solution, and a permanent red is obtained at the point of neutralization.

"The determination of magnesia proceeds as follows:

"Transfer the neutral solution to a large test tube 12 inches long and 1 inch inside diameter marked at 100 c. c. Heat to boiling and add 1-5 normal caustic soda solution, about one c. c. at a time, boiling for a moment after each addition until a deep red color is obtained which does not pale on boiling.

"This point can be easily recognized within one-half c. c. after a little practice. Note the number of c. c. soda solution added to the neutral solution as second alkali. Dilute to 100 c. c., boil for a moment and set the tube aside to allow the precipitate to

*"Cement and Engineering News," March, 1903.

settle. When settled take out 50 c. c. of the clear liquid by means of a pipette and titrate back to colorless with 1-5 normal acid. Multiply by 2 the number of c. c. of acid required to neutralize and note as second acid.

"Calculation:

"Second alkali: second acid $\times 2 \times 0.40 =$ per cent. magnesia. First acid $\times$ (1st alkali + 2d alkali — second acid) $\times 2 \times 0.56 =$ per cent. calcium oxide."

SUPPLEMENTARY.

The Detection of Adulterants.—The common adulterants of Portland cement are natural cement, limestone, cement rock, slag, cinder, sand and in foreign cements, hydraulic lime. Some of these, moreover, so nearly approach the chemical composition of cement that they may readily escape detection in ordinary analysis, so that recourse must be made to special methods in order to determine their character and amount.

The chemical tests most generally employed for this purpose are:—loss on ignition, weight of carbon di-oxide absorbed, and reduction of potassium permanganate.

The determination of loss on ignition has already been described,* and for a normal Portland cement should rarely exceed 2½ per cent. If much above that figure and the cement is not underburned, as shown by the specific gravity, it would indicate the presence of natural cement, a carbonate rock, or hydraulic lime.

The amount of carbon di-oxide absorbed is obtained by placing about 3 grams of finely ground material in a stream of the gas, then drying it over sulphuric acid and determining the increase in weight. Normal cement rarely absorbs over 0.05 per cent.; excess indicates natural cement or hydraulic lime.

The reduction of potassium permanganate test is made by treating one gram of finely ground cement with a mixture of 50 c. c. of dilute sulphuric acid and 100 c. c. of water and then titrating with a potassium permanganate solution of known strength. A gram of normal Portland cement should not reduce more than 3 milligrams of permanganate, while a gram of slag reduces from 45 to 75 milligrams.

The presence of cinder or sand may be detected readily by

*See page 186.

treating the cement with dilute (1:1) hydrochloric acid, the sand or cinder remaining as an insoluble residue, which may be examined to determine its character. The presence of slag also may generally be detected by the same treatment, due to the evolution of hydrogen sulphide gas which can be recognized by its characteristic odor or by placing over the vessel a filter paper moistened with lead acetate which will be turned black if this gas is present.

The specific gravity of Portland cement is much greater than any of its adulterants and this gives another method of detection. Portland cement averages a specific gravity of 3.15, natural cement 2.85, slag 2.85, limestone 2.60, sand 2.65, and cinder 2.70, so that a large amount of adulteration could readily be observed in the ordinary specific gravity test. However, on account of the many other conditions that also operate to produce a low specific gravity this test alone is never positive, but the difference in specific gravity between cement and its adulterants may be utilized in the following method devised originally by Le Chatelier:

This method consists in preparing a liquid with a specific gravity of about 2.95 by diluting iodide of methylene (sp. gr. 3.34) with benzole or turpentine and adding the cement, which sinks in the liquid while the adulterant floats on the surface. The liquid is conveniently prepared by placing about 12 c. c. in a small test tube with a crystal of aragonite which has a specific gravity of 2.95 and then slowly adding benzole with constant stirring until the liquid is neutral to the crystal so that it will neither sink nor float. It is then quickly transferred to the separatory funnel (Fig. 107) which should be about four-fifths full. One to two grams of the sample to be tested are then weighed and brushed into the liquid, stirred a few moments with the platinum rod, then tightly stoppered and set aside for about half an hour, while the separation takes place. By careful manipulation the two portions may be drawn off separately, caught on tared filters and after washing with benzole, dried and weighed, thus giving the relative amount of the adulteration, which may then be subjected to analysis and its character determined. Care must be taken in operating this apparatus to keep it tightly closed or the specific gravity of the liquid will rapidly increase due to the evaporation of the benzole.

The microscope affords another means for the detection of adulteration. It is best to employ a low power objective, about $\frac{2}{3}$ inch, and to examine that part of the cement which passes the No. 100 and is retained on the No. 200 sieve. The cement clinker can easily be recognized by its honeycombed appearance and its dark, almost black, color. Underburned clinker appears brownish and semi-transparent. Plaster of Paris appears soft and white. Slag is characterized by its grey color and angular fracture. Raw rock has about the same color as clinker, but lacks its honeycombed appearance. The debris of iron and

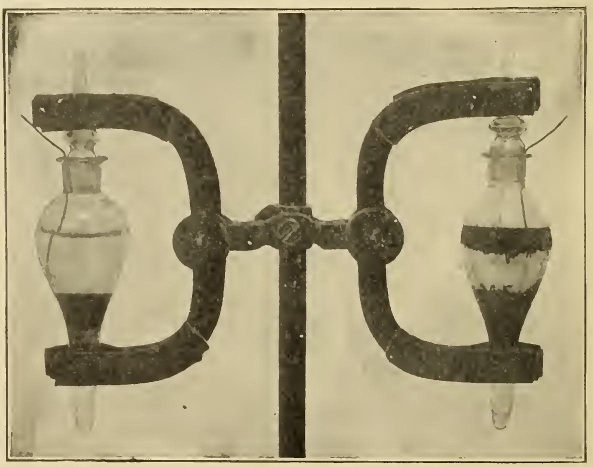


FIG. 107.—Apparatus for Detecting Adulterations by Separation with Methylene Iodide.

flint from the mills as well as particles of unburned coal may be readily recognized.

Equipment.—The following list gives the apparatus and chemicals necessary in a practical routine laboratory running an average of say four samples a day. The articles marked with an asterisk (*) should be increased or diminished for greater or less volume of work, while those marked with a dagger (†) are only required in determinations of carbonic acid and the alkalies or in tests for adulterations. The cost of the list as given should be from 250 to 300 dollars.

A small equipment adapted to the occasional testing of single samples for the common constituents will cost from 100 to 150 dollars.

APPARATUS.

- Chemical balance (sensible to 0.1 mg.).
- Rough balance (sensible to .1 g.).
- *4 platinum crucibles.
- †1 " Gooch crucible.
- †1 " dish—100 c. c.
- 1 2½-inch agate mortar.
- 1 small steel mortar.
- *2 5-inch desiccators.
- *12 4-inch casseroles.
- *12 No. 3 beakers.
- *12 No. 4 "
- *6 No. 6 "
- *6 No. 8 "
- *12 4-inch watch glasses.
- *12 6-inch " "
- *12 2-inch funnels.
- *12 3½-inch funnels.
- 3 50 c. c. burettes.
- 1 100 c. c. burette.
- 1 1,000 c. c. graduated cylinder.
- 2 100 c. c. graduated cylinders.
- 3 pipettes (10 c. c. — 25 c. c. — 50 c. c.).
- 2 1,000 c. c. volumetric flasks.
- 2 1 litre flasks.
- 2 500 c. c. flasks.
- 1 500 c. c. filter flask.
- 2 12 oz. Erlenmeyer flasks.
- †2 8 oz. " "
- †1 washing bottle.
- †1 $CaCl_2$ jar.
- †1 12-inch condenser.
- †4 8-inch U tubes.
- †2 6-inch U tubes (with ground glass stoppers).
- †1 2-oz. separatory funnel.
- †1 CO_2 bottle.
- 1 aspirator bottle or suction pump.
- †1 special separatory funnel (for adulterations).
- *1 doz. specimen bottles.
- 1 doz. 6-inch test tubes.
- *½ doz. No. 0 porcelain crucibles.
- 1 doz. rubber tips.
- 1 200° C. thermometer.
- 1 water still.
- 1 water bath.
- 1 hot plate (11 x 18).
- 1 drying oven.
- *4 Bunsen burners.
- *1 blast lamp and bellows.
- *6 retort stands.
- 2 clamps.
- 2 burette stands.
- *4 filter stands (double).
- 1 test tube rack.
- 12 clay triangles.
- 1 pair crucible tongs (nickel).
- 1 cork borer.
- * Glass rods and tubing.
- Wire gauze.
- Asbestos board.
- Rubber tubing.
- " stoppers.
- Corks
- Files.
- 2 small camel's hair brushes.
- 2 6-inch spatulas.

CHEMICALS.

- Hydrochloric acid.
- Sulphuric acid.
- Ammonia.
- Nitric acid.
- Oxalic acid.
- Hydrofluoric acid.
- Sodium carbonate.
- †Ammonium chloride.
- † " carbonate.
- † " oxalate.
- Microcosmic salt.
- Barium chloride.
- Zinc.
- Potassium permanganate.
- †Soda lime.
- Calcium chloride.
- †Potassium hydrate.
- " nitrate.
- " bisulphate.
- Sodium hydrate.
- †Copper sulphate.
- †Platinic chloride.
- Silver nitrate.
- Lead acetate.
- †Calcium carbonate.
- Alcohol.
- Iron wire.
- Phenol-phthalein.
- †Methylene iodide.
- †Benzole.
- Limestone.
- Iron sulphide.
- Qualitative filter paper, 9 cm.
- Ashless qualitative filter paper, 9 cm. and 11 cm.
- Ashless qualitative filter paper for sulphate, 7 cm.

It would scarcely be profitable to give descriptions of the various fixtures, hoods, sinks, water and gas arrangements, etc., necessary to equip a chemical laboratory, since each particular laboratory has different conditions to meet, and each chemist has individual preferences as to their arrangement. A convenient bath for running down solutions and evaporating silicas can be made by placing a steam coil in the bottom of a hood and covering it with 2 or 3 inches of sand. An automatic motor driven blast will also be found a great convenience and timesaver. The author has installed in the Philadelphia Laboratories a system of ovens and plates heated by electricity which have

TABLE XLVI.—Symbols and Atomic Weights of the Elements Entering into the Analysis of Cement.

(Atomic Weights Based on H = 1.000.)

Aluminum	Al	26.9	Magnesium	Mg	24.18
Arsenic	As	74.4	Manganese	Mn	54.6
Barium	Ba	136.4	Nitrogen	N	13.93
Bromine	Br	79.36	Oxygen	O	15.88
Cadmium	Cd	111.6	Phosphorous	P	30.77
Calcium	Ca	39.8	Platinum	Pt	193.3
Carbon	C	11.91	Potassium	K	38.86
Chlorine	Cl	35.18	Silicon	Si	28.2
Chromium	Cr	51.7	Silver	Ag	107.12
Hydrogen	H	1.00	Sodium	Na	22.88
Iodine	I	125.9	Sulphur	S	31.83
Iron	Fe	55.5	Tin	Sn	118.1
Lead	Pb	205.35	Zinc	Zn	64.9

given great satisfaction and are recommended where an electric current is convenient and cheap.

Value of Chemical Analysis.—Only the mills and the large private and permanent laboratories have need of a chemical laboratory for the testing of cement. In the mills, the control of the product rests entirely on the composition of the raw materials and there a laboratory is a positive necessity. From the consumer's standpoint it is only necessary to know that the injurious constituents, principally magnesia and sulphuric acid, are within allowable limits. With the magnesia little trouble is ever encountered since none of the domestic cements average over 3 or 3½ per cent., which is entirely safe for all ordinary construction. The content of sulphuric acid should be checked from time

TABLE XLVII.—Factors for Use in Cement Analysis, With Their Logarithms.

(Factors Based on Atomic Weights Given in Table XLVI.)

Found	Sought	Factor	Logarithm	Found	Sought	Factor	Logarithm
$Ba\ SO_4$	$Ca\ SO_4$	0.5831	9.76574	$Ca\ SO_4$	$Ca\ O$	0.4120	9.61490
$Ba\ SO_4$	$Ca\ SO_4\ 2H_2O$	0.7374	9.86770	$Ca\ SO_4$	$Ca\ SO_4\ 2H_2O$	1.2645	0.10192
$Ba\ SO_4$	S	0.1373	9.13767	$Ca\ SO_4$	SO_3	0.5880	9.76938
$Ba\ SO_4$	SO_3	0.3429	9.53517	SO_3	$Ca\ SO_4$	1.7006	0.23060
$Ba\ SO_4$	$H_2\ SO_4$	0.4206	9.62387	$Fe_2\ O_3$	$Fe\ O$	0.8999	9.95419
$Ba\ SO_4$	$Ca\ S$	0.3091	9.49010	$Fe\ O$	$Fe_2\ O_3$	1.1112	0.04579
$Ba\ SO_4$	$Fe\ S_2$	0.2570	9.40993	$Fe_2\ O_3$	$Fe\ S_2$	1.5022	0.17673
$Cd\ S$	$Ca\ S$	0.4994	9.69845	$Fe\ S_2$	$Fe_2\ O_3$	0.6656	9.82321
$Cd\ S$	S	0.2219	9.34616	$Fe\ S_2$	Fe	0.4657	9.66811
$Cd\ S$	$Ca\ SO_4$	0.9422	9.97414	$Fe\ S_2$	S_2	0.5342	9.72770
$Ca\ O$	$Ca\ CO_3$	1.7843	0.25147	$Mg\ O$	$Mg\ CO_3$	2.0901	0.32017
$Ca\ O$	$Ca\ SO_4$	2.4272	0.38511	$Mg\ CO_3$	$Mg\ O$	0.4784	9.67979
$Ca\ O$	$Ca\ SO_4\ 2H_2O$	3.0695	0.48607	$Mg_2\ P_2\ O_7$	$Mg\ O$	0.3624	9.55919
$Ca\ CO_3$	$Ca\ O$	0.5604	9.74850	$Mg_2\ P_2\ O_7$	$Mg\ CO_3$	0.7575	9.87938
$Ca\ CO_3$	$Ca\ SO_4$	1.3603	0.13363	$K_2\ Pt\ Cl_6$	KCl	0.3071	9.48728
$Ca\ CO_3$	$Ca\ SO_4\ 2H_2O$	1.7202	0.23558	$K_2\ Pt\ Cl_6$	$K_2\ O$	0.1941	9.28803
$Ca\ CO_3$	CO_2	0.4395	9.64296	KCl	$K_2\ O$	0.6320	9.80072
CO_2	$Ca\ CO_3$	2.2750	0.35698	$Na\ Cl$	$Na_2\ O$	0.5308	9.72493

to time and not allowed to exceed 1.75 or at most 2 per cent., but the best method for the consumer, unless he has a permanent laboratory, is to send occasional samples to one of the many private laboratories for an analysis of sulphuric acid and occasionally for magnesia, which will cost but 2 or 3 dollars a sample, and thus will effect a great saving over the cost of making

TABLE XLVIII.—For Reducing Values of $Ba\ SO_4$ to SO_3—Based on the Factor 0.343.

	0.00	0.01	0.02	0.03	0.04
0.00	0.0000	0.0034	0.0069	0.0103	0.0137
0.10	0.0343	0.0377	0.0412	0.0446	0.0480
0.20	0.0686	0.0720	0.0755	0.0789	0.0823
0.30	0.1029	0.1063	0.1098	0.1132	0.1166
0.40	0.1372	0.1406	0.1441	0.1475	0.1509
0.50	0.1715	0.1749	0.1784	0.1818	0.1852
0.60	0.2058	0.2092	0.2127	0.2161	0.2195
0.70	0.2401	0.2435	0.2470	0.2504	0.2538
0.80	0.2744	0.2778	0.2813	0.2847	0.2881
0.90	0.3087	0.3121	0.3156	0.3190	0.3224
1.00	0.3430	0.3464	0.3499	0.3533	0.3567

	0.05	0.06	0.07	0.08	0.09
0.00	0.0171	0.0206	0.0240	0.0274	0.0309
0.10	0.0514	0.0549	0.0583	0.0617	0.0652
0.20	0.0857	0.0892	0.0926	0.0960	0.0995
0.30	0.1200	0.1235	0.1269	0.1303	0.1338
0.40	0.1543	0.1578	0.1612	0.1646	0.1681
0.50	0.1886	0.1921	0.1955	0.1989	0.2024
0.60	0.2229	0.2264	0.2298	0.2332	0.2367
0.70	0.2572	0.2607	0.2641	0.2675	0.2710
0.80	0.2915	0.2950	0.2984	0.3018	0.3053
0.90	0.3258	0.3293	0.3327	0.3361	0.3390
1.00	0.3601	0.3636	0.3670	0.3704	0.3739

these tests himself. A few simple reagents and test-tubes will be sufficient for making the adulteration tests just described, but that need be the only outfit required. The complete analyses made by the permanent laboratories are more a matter of record and experiment, than of value in the acceptance of material.

TABLE XLIX.—For Converting $Mg\ P_2\ O_7$ to Mg O—
Based on the Factor 0.362.

	0.00	0.01	0.02	0.03	0.04
0.00	0.0000	0.0036	0.0072	0.0109	0.0145
0.10........	0.0362	0.0398	0.0434	0.0471	0.0507
0.20........	0.0724	0.0760	0.0796	0.0832	0.0868
0.30........	0.1086	0.1122	0.1158	0.1195	0.1231
0.40........	0.1448	0.1484	0.1520	0.1557	0.1593
0.50........	0.1810	0.1846	0.1882	0.1919	0.1955
0.60........	0.2172	0.2208	0.2244	0.2281	0.2317
0.70........	0.2534	0.2570	0.2606	0.2643	0.2679
0.80........	0.2896	0.2932	0.2968	0.3005	0.3041
0.90........	0.3258	0.3294	0.3330	0.3367	0.3403
1.00........	0.3620	0.3656	0.3692	0.3729	0.3765

	0.05	0.06	0.07	0.08	0.09
0.00........	0.0181	0.0217	0.0253	0.0290	0.0326
0.10........	0.0543	0.0579	0.0615	0.0652	0.0688
0.20........	0.0905	0.0941	0.0977	0.1014	0.1050
0.30........	0.1267	0.1303	0.1339	0.1376	0.1412
0.40........	0.1629	0.1665	0.1701	0.1738	0.1774
0.50........	0.1991	0.2027	0.2063	0.2100	0.2136
0.60........	0.2353	0.2389	0.2425	0.2462	0.2498
0.70........	0.2715	0.2751	0.2787	0.2824	0.2860
0.80....... .	0.3077	0.3113	0.3149	0.3186	0.3222
0.90........	0.3439	0.3475	0.3511	0.3548	0.3584
1.00........	0.3801	0.3837	0.3873	0.3910	0.3946

CHAPTER XII.

SPECIAL TESTS.

The tests considered in this chapter are employed but rarely in ordinary routine, and have little, if any, importance, so far as the customary reception tests are concerned. Only sufficient will therefore be given to enable the operator to understand the reasons for their occasional employment and the common methods of making the determinations.

Compression Tests.—These tests usually are made for the comparison of different sands and stones intended for use in concrete, and for other purposes in which the concrete itself must be tested, and not the cement or mortar composing it. The test is frequently made for experimental purposes on cements or mortars, but rarely for purposes of reception.

For tests of concrete, compression tests are the most suitable for the reason that either tensile or transverse specimens must be of such a size, if reliable results are desired, that they are very unwieldy and awkward to handle. A 6-inch cube, however, is sufficient for tests in compression.

The common form of specimen is that of a cube, 2 ins. on a side for mortars, and 6 ins. for concrete. The material fails generally by pushing out the sides laterally leaving pyramidal pieces in the middle, the failure being in the nature of a shear along surfaces inclined about 30° to 35° with the vertical. Since the material fails at this angle, a cube evidently will not give theoretically true results, but its employment is so universal that it would be difficult to institute a change. Cylinders are often employed instead of cubes, because they can be filled more uniformly, it being very difficult to thoroughly compact the corners of the cube. The broken halves of briquettes are also tested occasionally, but because of their small depth in comparison with the area they give abnormally great results. Johnson* states that half-briquettes should be multiplied by the correction factor 0.83 to make the results obtained from them and from cubes

*"The Materials of Construction," by J. B. Johnson.

comparable. For certain tests the author has used small cylinders 1 in. in area and 1 in. high with very satisfactory results.

The bearing surfaces of the specimens must be carefully dressed to true planes before test, and it is advisable to have one surface bearing on a ball and socket joint to correct for any slight angle between the planes of the surfaces. To take up small irregularities, it is common to rest the block on blotting paper, sheet lead, or plaster of Paris. When plaster is employed it is gauged, put between sized paper at both top and bottom of the specimen, on which a very low stress is placed, the plaster setting while the cube is in position. The author uses three thicknesses of blotting paper at the top and bottom of each cube,

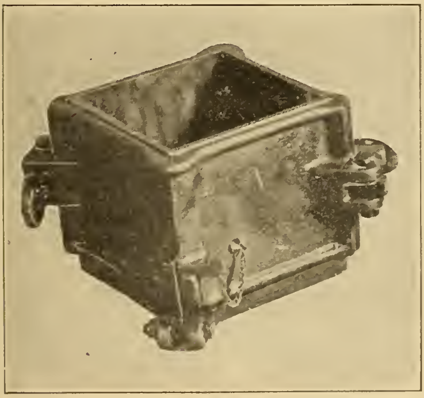


FIG. 108.—Mould for 6-in. Concrete Cubes.

FIG. 109.—Mould for 6-in. Cylinders of Concrete.

and finds this surface most satisfactory for rapid and accurate work. The results obtained, when any of these cushions are used, will, however, be slightly lower than those from cubes having a direct bearing on the steel plates. For dressing the surfaces a machine like that shown in Fig. 117, page 220, may be used to advantage.

For 6-in. cubes of concrete, a good form of mould made of cast iron, is shown in Fig. 108. Figures 109 and 110 give forms of cylindrical moulds, the latter being an inexpensive form made of sheet iron held by a clamp. The 2-in. cube moulds are made either singly or in gangs (Fig. 111).

The machines usually employed for tests of concrete are the so-called "universal" machines which can be made adaptable for tests of tension and cross-breaking, although entirely too cumbersome for making either of these tests on small briquettes or prisms of cement. A type of these machines is shown in Fig. 112; power is necessary to operate them satisfactorily. For breaking 6-in. cubes of the richer mixtures of concrete, a capacity of 150,000 pounds is necessary, and even this amount is occasionally exceeded, although most concrete will fail under 100,000 pounds. For 6-in. cylinders, 100,000 pounds will usually be sufficient.

FIG. 110.—A Simple Form of Cylindrical Mould for Concrete.

For tests of 2-in. cubes a similar "universal" machine of smaller capacity may be employed, or a special machine such as is illustrated in Fig. 113. In this, the load is due to hydraulic pressure applied by the hand-wheel at the side, while its amount is read on the gauge. This particular machine is of 30,000 pounds capacity. Hydraulic machines, however, are generally less accurate and satisfactory than those in which the load is supplied through direct gearing. The compression attachment furnished with the long-lever cement machines is convenient for testing one-inch cubes and cylinders of mortar. A 150,000 pound universal machine, and a long-lever cement machine with attachments for compression and transverse tests will be sufficient to make all strength tests of cements, mortars and concretes.

Strength in Compression.—The ratio of compressive strength determined from cubes or cylinders to tensile strength as determined from the standard briquettes will vary all the way from 3 to 15, depending on the character of the specimens, their age, condition, richness, and method of treatment. The average ratio varies from 5 to 10. Johnson* gives

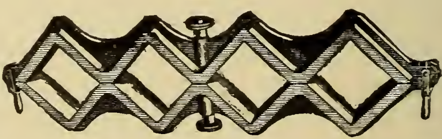


FIG. 111.—Gang Mould for 2-in. Cubes.

*"The Materials of Construction," by J. B. Johnson.

the following formula for this ratio based upon tests by Tetmajer on 1 : 3 mortar:

$$\text{Ratio of } \frac{\text{compressive strength}}{\text{tensile strength}} = 8.64 + 1.8 \log A$$

where A = the age in months. This gives a ratio of 8.6 for one

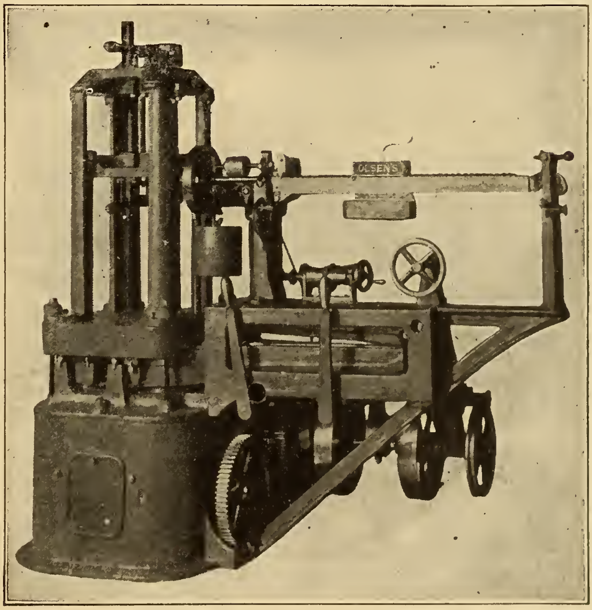


FIG. 112.—The Olsen Universal Testing Machine.

month, to 10.6 for 1 year, which is high for this mixture. The ratio generally increases both with age and with the richness of

TABLE L.—Showing the Relation of the Strengths of Cement Under Different Forms of Stress.

(From "Cements, Mortars and Concretes," by M. S. Falk.)

Age in Weeks	Mixture	Tension, Ult. Resistance Pounds per Square Inch		Compression, Ult. Resistance Pounds per Square Inch		Bending, Extreme Fibre Stress in Lbs. per Sq. Inch		Shear, Ult. Resistance Pounds per Square Inch	
		Specimens Hardened in							
		Air	Water	Air	Water	Air	Water	Air	Water
1	1 : 0	231	224	1860	1910	695	625	276	271
	1 : 3	106	95	920	880	273	247	109	116
	1 : 5	68	64	543	537	168	158	81	77
4	1 : 0	266	294	2460	2490	860	887	316	346
	1 : 3	148	169	1500	1040	392	381	182	181
	1 : 5	119	103	962	977	284	276	136	131
104 to 113	1 : 0	257	292	3400	4680	1010	1350	388	415
	1 : 3	244	272	2080	3340	748	973	294	375
	1 : 5	177	232	1510	2960	545	810	248	364

Note.—Each value average of 9 tests.

the mortar. Table L. gives a compilation by Falk* of the results of tests made by Bauschinger, and shows the relation between the strengths under different forms of stress; each value represents 9 tests. The ratio in this table varies from 13.2 to 8.0, for specimens kept in air, and from 16.0 to 7.3 for those in water. These values are on the whole typical, although the maximum is abnormal.

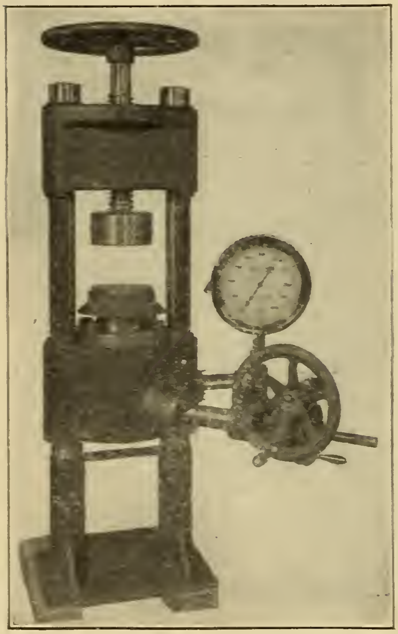


Fig. 113.—Hydraulic Machine for Compression Tests.

Transverse Tests. — It has frequently been urged that transverse instead of tensile tests be adopted for ordinary routine, on account of the simpler machines required, and the doing away with the tensile clip which is far from accurate. The great objections to them, however, are the larger size of the specimen, the fact that the least imperfection or chip near the center makes them almost worthless, and that greater uniformity or homogeneity is required. For experimental purposes and for obtaining approximate data on strength, however, they are often employed.

The size of specimen most frequently tested is a prism one inch square and either six or twelve inches long; specimens two inches square are less often made. The standard of the French Commission is two centimeters square and 12 centimeters long.

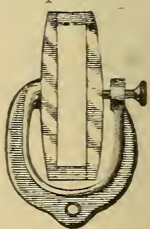


Fig. 114. — Mould for Prisms of Mortar for Transverse Test.

A common form of mould is shown in Fig. 114, which may be improved by the use of end clamps instead of the cumbersome arrangement given. Two forms of gang moulds for transverse prisms are illustrated on pages 231 and 232.

*"Cements, Mortars and Concretes," by M. S. Falk.

For testing these prisms, the attachments furnished with the long lever cement testing machines will be found most convenient. For rough tests three knife edges and a pail to hold sand or water are sufficient.* The knife edges should be rounded rather than sharp to prevent local crushing, and the prisms should be broken on their sides, so that the variation of the upper surface is minimized.

The results of transverse tests are customarily expressed by means of the "modulus of rupture," which theoretically is the tensile stress on the extreme fibre of the specimen, provided the material has not been stressed beyond its elastic limit. When extended to the point of rupture, the formula no longer holds, so that its use in the expression of ultimate values is purely empiric, and for this reason the ratio between tensile and transverse strength is not constant, but varies with certain conditions. The modulus of rupture is written $M = \frac{3\,w\,l}{2\,b\,h^2}$, in which w = the center load, l = the span, b = the width and h = the depth of the specimen. To simplify calculations prisms one inch square are often tested on a span of $6\frac{2}{3}$ ins., in which case the center load equals one-tenth of the modulus of rupture; two-inch prisms on a $5\frac{1}{3}$-inch span have the center load and modulus equal.

The ratio between transverse and tensile strength varies from about 1.3 to 2.5. Generally, it varies inversely with the span, and directly with the cross-section, age and richness of the mortar. Durand-Claye, testing neat Portland cement on the standard specimen of the French Commission, found the ratio to be 1.92 at 7 days and 1.86 at 28 days. The ratios given in Table L. vary from 2.25 to 4.65 and increase with age; these values are exceptionally high. A series of tests by the author on specimensof 1:3 mortar are given on page 235. The accuracy of carefully made tensile and transverse tests is about equal.

Tests of Adhesion.—That a cement or mortar should be capable of adhering to an inert material is as important for purposes of construction as its cohesive properties, and yet this test is made only infrequently and then more for experimental research than for practical purposes. The tests are commonly made to determine the adhesive strength of cement pastes and mortars

*For description of rough transverse testing machines, see page [illegible] to stone, metal, or to hardened mortar. The form of mould as recommended by the French Commission on Standard Methods of Testing is shown in Fig. 115. For the test, an adhesion block of a fine cement mixed with two parts of sand is first made in a special mould in the form of a half briquette, when hardened the adhesion surface is polished with emery paper, then fastened into the mould and the other half filled with the cement to be tested, Adhesion blocks of stone and metal are also employed.

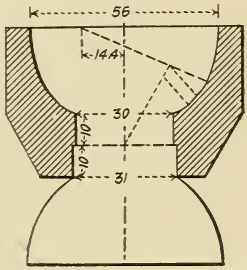


Fig. 115.—Form of Adhesion Mould and Block Recommended by the French Commission. Reproduced from Spalding's Hydraulic Cement

These tests may be made in an ordinary mould filling half with a rich standard mortar and when hardened filling the other half with the mortar to be tested. A small block of iron in the center will give a smooth surface to the first mortar. Tests of adhesion to metal or stone may be simply made by preparing small pieces $1 \times 1 \times \frac{1}{4}$ in. of the adhesion surface material and placing them in the center of the briquette mould filling it on

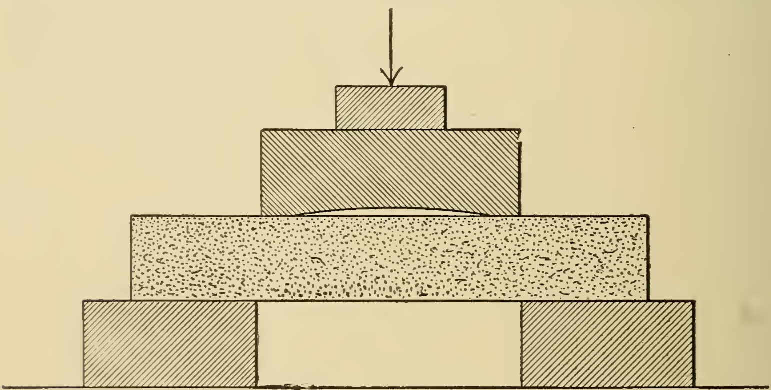


Fig. 116.—Illustrating the Method for Conducting Shearing Tests.

both sides with the mortar to be tested. When many of these tests are to be made, it is convenient to cut grooves in the sides of the mould and make the plates slightly over an inch in width thus holding the plate firmly and accurately in the center.

Adhesive strength, like cohesive, is subject to the same variations due to consistency and richness of the mortar, age, environment, method of treatment, etc. The strength also increases with the roughness and porosity of the surface to which it is tested. The retempering of mortar is said to affect the adhesive far more than the cohesive strength; Chandlot states that this reduction may amount to 50%.

The adhesion of cement mortar (1 : 2) to sandstone at 28 days averages about 100 pounds, to ground glass about 50 pounds, to iron from 50 to 75 pounds.

Shearing Tests.—Although mortars and concretes are frequently subjected to shearing stresses, tests for shearing strength are but seldom made. The simplest method of making these tests is shown by the diagram in Fig. 116. The upper bearing should be slightly arched to avoid the introduction of other stresses and the load applied exactly in the center. A convenient specimen to test is a prism 1 × 1 × 6 ins., the distance between the upper bearings being 3 ins. and between the lower about 3 1-16 ins., so that the upper bearing comes slightly within the lower. The total load is then twice the shearing strength in pounds per square inch. Tests are sometimes made by cementing three bricks together, the middle one projecting above the other two, and applying the load on the middle brick, but this method is liable to be inaccurate on account of the introduction of both adhesive and transverse stresses.

From Table L., the ratio of shearing to tensile strength is seen to vary between 1.03 and 1.57. Mesnager gives the ratio as 1.2 to 1.3. Tests by the author seemed to show a ratio but slightly over unity, but it may generally be considered safe to allow a shearing value of 1.2 times the tensile strength. On account of the frequent designing of structures to withstand shearing stresses, more data on this subject are much to be desired.

Abrasion Tests.—These are made by forming a block of mortar or concrete and placing it in contact with a grinding surface under a definite pressure. A machine made by Riehle Bros. for this purpose is shown in Fig. 117. It consists of a revolving cast iron disc, on which the specimen rests, while the pressure on it can be regulated by means of movable weights on the long lever. Sand and water are automatically fed between the specimen and the disc. The test consists of determining the amount of wear produced after the specimen has been subjected to a given number of revolutions of the disc, under a definite pressure.

The test has a certain value for determining the relative abrasive properties of cement mixtures intended for use in sidewalks, floors, and other similar purposes. Sand mortars give better abrasion tests than neat pastes. Mr. E. C. Clarke* found that a mixture of 1 part Portland cement to 2 parts of sand gave the best resistance to abrasion.

Porosity.—The porosity of a mortar or concrete is the amount of void space in the hardened specimen, and is usually expressed

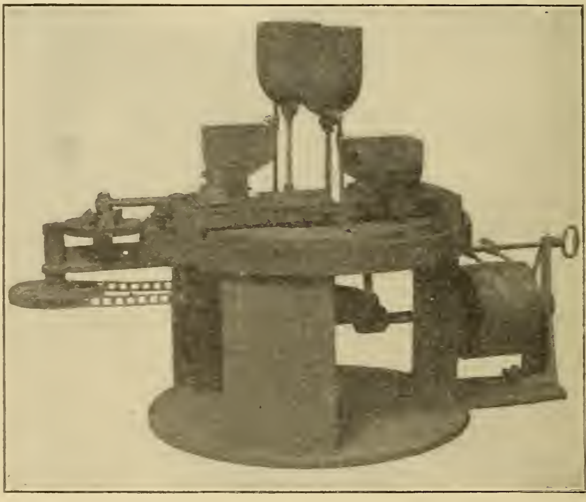


FIG. 117.—Machine for Testing the Abrasive Qualities of Cement.

as the percentage of voids to the total volume. The determination consists, therefore, of the measurement of the cubical contents of the specimen and the actual volume of its ingredients. The measurement of the specimen is simple if it be of regular form; if not, it is weighed in water and again in air after it is completely saturated, the difference between these two weights divided by the weight of a cubic foot of water giving the apparent volume. Grease is sometimes rubbed on the specimen to prevent it changing its saturation and hence its weight during the process of weighing. The actual volume of the ingredients

*Transactions, American Society of Civil Engineers, Vol. XIV., p. 167.

is obtained in the same manner by weighing the dried specimen in air, and after saturation in water. The test pieces are dried at a temperature of 110° Fahr., weighed, then immersed in water until completely saturated, weighed in water, and then again in air. Complete saturation is effected either by exhausting the air by placing the specimen in water under a pump, or by boiling it, the former method being preferable. The porosity is then $\frac{D - W}{P - W}$, where D = weight air dry, W = weight in water after saturation and P = weight in air when saturated. The standard size of specimen for this test is a 3-inch cube, while the standard test is made on mortar (1:3) at the age of 28 days. The porosity of normal testing mortar averages from 20 to 35 per cent. This test is never employed for purposes of reception.

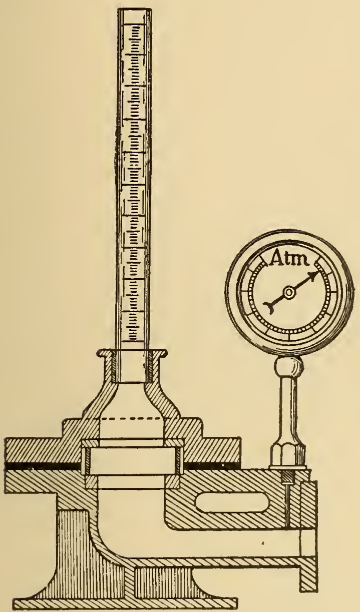


FIG. 118.—Tetmajer's Apparatus for Testing Permeability.

Permeability.—The permeability of a mortar is the rate with which water under a fixed head will pass through it under definite conditions. This must not be confused with the test of porosity, which is a determination of void space, while permeability determines the rate at which water passes through those voids. There is no fixed ratio between the two properties.

Figure 118 shows the apparatus of Prof. Tetmajer in which the specimen is made in the form of a disc, mounted in rubber cushions and subjected to a definite water pressure, the rate at which the water rises in the tube being a measure of the permeability.

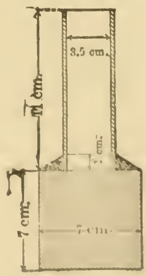


FIG. 119

Apparatus for Testing Permeability. Recommended by the French Commission

Figure 119 illustrates the device recommended by the French Commission on Methods of Testing, which is merely a cube of mortar to which a circular glass tube is cemented with a paste of neat cement. A rubber hose is fastened to the tube and connected with a reservoir of water at a height of 4 inches, 3 feet 3 inches, or 33 feet (0.1, 1.0, or 10.0 meters) above the specimen, according to its degree of permeability. The test consists in measuring the amount of water passing through in an hour. The standard specimen is a 2 or 3-in. cube of 1:3 mortar, 28 days old, and which has been immersed in water for at least 48 hours prior to the test. It is also advisable to allow the block to remain immersed during the determination.

A rough test to determine the comparative permeability of concretes may be made in the same manner, by cementing a one-inch iron pipe to a cube or disc of the concrete. This test, like that of porosity, is only made for purposes of experiment, and never for reception.

NOTE:—For data on porosity and permeability, the reader is referred to: R. Feret "Sur la Compacité des Mortiers Hydrauliques" in "Annales des Ponts et Chaussées" Vol. IV, 1892; L. von Tetmajer in "Methoden und Resultate der Prüfung der Hydraulischen Bindemittel" (Zurich, 1893); E. Chandlot—"Ciments et Chaux Hydrauliques"; Report of the French Commission on Methods of Testing the Materials of Construction, Vol. I, 1894, and Vol. IV, 1895; S. E. Thompson, Proceedings Am. Soc. of Civil Engineers, Aug. 1903, pp. 648-649, from which cut 119 was taken; and F. W. Taylor and S. E. Thompson in "Concrete—Plain and Reinforced."

Yield Test of Mortar.—This test is occasionally employed to determine the economy of different mixtures and consistencies of pastes and mortars. The yield of a mortar is the volume occupied by a paste or mortar gauged to a given consistency and made from a unit weight of cement or a mixture of cement and sand. The test is made by weighing 1,000 grams of cement or sand mixture, gauging it with a fixed percentage of water and introducing it into a graduated cylinder, in such a manner as to avoid the presence of air, and observing the volume occupied.

Another method of making this test is to form the paste into a block and after greasing the surfaces, to obtain its volume by weighing in air and suspended in water, or by actual displacement.

TESTS OF SAND.

The testing of sand for use in mortar is so closely allied to that of cement that a brief mention of the methods employed to determine the relative value of different sands may well be made. The common tests employed for this purpose are its fineness, its degree of purity, the character of the grain, the specific gravity, the percentage of voids, and strength tests in comparison with a standard sand.

The fineness is tested by sifting through a series of sieves, such as the Nos. 10, 15, 20, 30, 40, 60, 74, 100, 150 and 200.* A mechanical shaker, such as is shown in Fig. 34, page 72, is convenient for this purpose. The results are usually expressed as the percentage by weight passing each sieve. The size of sand is frequently indicated by its uniformity coefficient, which is the ratio of the diameter of those particles which have 60% of the sand smaller than itself, to the diameter of those which have 10% smaller. †"Sand having a coefficient of over 4.5 is a good coarse sand for concrete work, and in comparing different natural sands the one having the highest uniformity coefficient may be considered the best."

The purity may be ascertained first by chemical analysis and secondly by the determination of the presence of loam, clay, or similar extraneous materials by elutriation methods. Elutriation consists of placing a weighed quantity of sand in a beaker or similar vessel, adding water, stirring it vigorously, and decanting off the material remaining in suspension after 15 seconds standing, repeating the process until the water pours off clear, then drying and weighing the residue. The effect of these impurities on the mortar depends both upon their own character and that of the sand, and also upon the richness of the mortar. The consensus of opinion seems to be, however, that generally organic loam is deleterious, while small percentages of clay or finely divided mineral matter are beneficial.

The character of grain is examined under a large reading glass, or microscope of low power. A rounded grain will give a denser mortar than an angular one, because it may be compacted more easily and hence has a lower void space. This is well shown by comparison between the two standard testing

*These sizes are recommended by Mr. W. B. Fuller.

†From "Concrete-Plain and Reinforced," by F. W. Taylor and S. E. Thompson.

sands—Ottawa and crushed quartz. The clause that sand should be sharp is a feature of many specifications, based upon a misconception or misunderstanding of this principle.

The specific gravity may readily be determined in the same manner as cement,* or less accurately in a tall graduated glass cylinder by first filling it half full of water then introducing a weighed quantity of sand, a few grains at a time to eliminate air bubbles, and noting the displacement; the specific gravity being the weight of the sand divided by the computed weight of the displaced water. The sand should be dried at a temperature of 212° Fahr., before making this test. Sand has an average specific gravity of 2.65.

The determination of voids is made by filling a measure with sand, and then weighing its contents. The sands tested should all be filled in the measure in an exactly similar manner either loose, shaken or compacted, since the void space is appreciably affected thereby. A tall 1,000 c. c. cylinder is a convenient measure, and can be filled to the upper mark very accurately. The weight of the sand in grams divided by 1,000, or the volume in c. c., then gives the net weight of the sand per c. c., and this divided by its specific gravity (2.65) and multiplied by 100 gives the percentage of voids. The determination is made sometimes by ascertaining the amount of water that can be introduced in a measure filled with sand, but this method is very inaccurate, both on account of the absorption of water by the sand, and on account of the great difficulty of eliminating air bubbles.

Strength tests are made by testing mortar briquettes in tension in comparison with standard sand or another sand of known value. Although the ratio of tensile strength to that of compression, cross-breaking, shearing and adhesion, varies considerably with different sands, the tensile test is usually considered sufficient for purposes of comparison. It is advisable to make other tests, however, particularly in compression and cross-breaking, if the facilities permit.

TESTS OF STONE.

The tests applied to stone are practically similar to those made on sand. For determining the size, sieves of 0.10, 0.15,

*See page 58.

0.20, 0.30, 0.45, 0.67, 1.00, 1.50, 2.25, and 3.00 ins.* may be used.

The specific gravity may be obtained in a manner similar to that employed for sand, or by weighing pieces of the stone in air and then suspended in water, the weight in air divided by the loss of weight in water giving the specific gravity.

Voids are determined best in a cubic foot measure in the same manner as sand. The percentage of voids is then the weight in pounds necessary to fill the measure, multiplied by 100, divided by the product of 62.3, the weight of water per cubic foot, times the specific gravity of the stone.

The comparative values of stone in compression are determined both by crushing small cubes or cylinders prepared from the stone itself, and by tests of concrete made with it. Tests in compression are the only ones generally employed.

*These sizes are recommended by Mr. W. B. Fuller.

CHAPTER XIII.

APPROXIMATE TESTS.

This chapter deals with tests made not strictly in accordance with orthodox methods, but by means of which it is possible to obtain information as to the quality of cement with little or no apparatus and without much experience in testing. Such tests are useful to the expert when examining material in a locality far removed from laboratories and apparatus, but are of especial value to the engineer of the small town, who uses but little cement, but wishes to know that what he does permit to enter his work is of the best quality. To send samples of the material to a distant laboratory frequently causes unnecessary delays, is somewhat expensive, and is more or less unsatisfactory, for the reason that the samples in transit may be subjected to conditions which will appreciably alter their physical properties, and also because the method of testing cannot then be supervised and, unless an elaborate report be submitted, it is difficult to know in what light to interpret the results. Generally speaking it is possible to obtain a very fair idea as to the quality of the cement by the use of the following tests, but on account of the inferior degree of accuracy they should always be interpreted rather liberally, or the material given the benefit of the doubt. Material positively unfit for service may, however, always be discovered by their use.

These approximate tests cover those of fineness, time of setting, strength, soundness and purity. Two tests that cannot be employed are quantitative chemical analysis and specific gravity, but for the small consumer the results of these tests are comparatively unimportant, and unless made with accuracy are apt to lead to misleading and erroneous conclusions.

Fineness.—For determining the fineness of cement by a rough method, the employment of the No. 100 sieve alone is recommended. It is true that there is no fixed relation between the results obtained on the No. 100 and No. 200 sieves, but nevertheless the average ratio is fairly constant, so that for this test it can be assumed with a fair amount of accuracy that the residue left on the No. 100 sieve is about a third of that on the

No. 200. For ordinary testing, the finer sieve is the more important, but, on the other hand, the greater part of the difficulties and irregularities of the determination lie in the obtaining and use of this sieve. Furthermore, it may be taken for granted that the manufacturers will not change the process of grinding because a comparatively small shipment is not to be tested on the finer sieve.

For the test, therefore, a No. 100 sieve alone is to be procured; neither a cover nor pan is necessary. It should be purchased from a reputable manufacturer who will guarantee the wire to be of proper diameter, while the average mesh per inch in both directions should be determined by actual count, and if not averaging between 96 and 100 the sieve should be returned. For this counting, a linen tester's microscope with a half-inch opening, such as is shown in Fig. 30, page 68, should be used. It may be purchased for 25 cents and will be found useful not only for this purpose, but also as a small pocket magnifying glass.

For the weighing, a chemist's or druggist's balance is advantageous, but an ordinary postal scale, weighing to 4 ounces, and showing a quarter-ounce clearly, may be employed.

Best practice in specifications limits the residue retained on the No. 100 sieve to 8%, and that on the No. 200 to 25%. Assuming that the first residue is one-third of the other, if it is known that less than 8% or one-twelfth of the cement is retained on the No. 100 sieve, the cement may be taken as sufficiently fine.

The test is made by weighing out 3 ounces of cement, placing it in the sieve, and shaking it over a piece of wrapping paper until only a few grains pass through after half a minute's sifting. From 10 to 12 minutes will generally suffice. The speed of sifting may be accelerated by placing half a dozen small coins in the sieve, which can be removed from the residue with ease. After sifting is complete, the residue on the sieve is shaken out on a clean piece of paper and brushed on the pan of the scales. If weighing less than ¼ of an ounce the cement is satisfactory. The accuracy of the test may be increased by weighing out 6 ounces originally, sifting about half at a time, and then weighing the combined residues which should not exceed half an ounce.

Elutriation methods are occasionally employed for rough tests of fineness, but except in the hands of the expert their resuts are apt to be very inaccurate, so that for the average engineer their use is not advised.

Setting.—For this test procure a pair of scales weighing a pound with comparative accuracy,* a druggist's graduate of a capacity of 8 fluid ounces, and an ordinary wash basin of china or paper, the former being preferable. Weigh out one pound of cement and measure $3\frac{1}{4}$ ounces of water. Place the cement in the basin, form it into a crater, pour the water in the center and knead it vigorously for a minute and a half in accordance with the method given for making briquettes on page 120. At the end of this time the cement should be nearly of the normal consistency, as obtained by the ball method,† or of such consistency that a ball of the paste about 2 ins. in diameter formed by rolling in the hands, dropped from a height of two feet will not crack badly, nor flatten more than half.‡ If the consistency of the paste is too wet or too dry, repeat the process using more or less water until the proper degree of plasticity is obtained. If the test is made at a place where the scales and measure cannot be procured, this consistency may be experimentally obtained after a few trials.

When a paste of proper plasticity has been made, form it rapidly into a rounded cake about the size of the cans in which shoe-blacking is usually furnished. One of these cans may actually be used to form the specimen if desired. The cake should be placed on a small piece of glass or metal, smoothed on the surface, and set aside in a cool place protected from draughts of air and direct sunlight.

Conservative specifications limit initial set to twenty minutes, so at the expiration of that period, the time being taken from the moment the water is poured on the cement, the cake must be examined. If the surface appears and feels wet, and the cake can still be worked slightly without cracking, the requirement is fulfilled. If, however, the surface appears dry and earthy, and an attempt to work cracks it, the material has failed. Cement setting in less than 20 minutes usually heats up considerably, so if the specimen feels warm during any portion of this period, it can be considered to fail in initial set.

*The postal scales used for the fineness test may be used by weighing out four ounces four times.

†See page 93.

‡A slight departure from this consistency is immaterial in this rough test.

At the end of 10 hours, the cake should be re-examined and at that time should have become so thoroughly hardened that a firm pressure of the thumb nail, or a pencil point, will not make an appreciable indentation. If it is not hardened, but still soft, the material has failed in hard set, and in construction may set up so slowly that it will seriously interfere with the progress of the work.

This rough examination will probably be more satisfactory and even more accurate, for a person unaccustomed to cement testing, than a test with such apparatus as the Gillmore needles which are easily mishandled, thus giving false results.

Strength—By the Tensile Test.—It is believed that but two things should be done with the tensile test—do it right, or leave it alone. There is room for so many inaccuracies in the conduct of this determination that when rough methods are employed, the results are apt to be very far from true, but the fact that the tensile test has been made in spite of many difficulties, tempts the operator to assign to them an undue amount of accuracy, whereas if other tests are made, known to be only approximate, they are seldom taken for more than they are worth.

The test in tension, even by approximate methods, cannot be made in a manner differing essentially from that given in Chapter IX., although some of the appliances may be simplified. Two things that should never be altered, however, are the moulds and the clips, for if any other form is employed, the values will be very different from those obtained under standard methods. The mixing of the briquettes may be performed in a wash basin, the appliances and method being similar to that already given under "setting," except that the quantity should be two pounds for neat cement, and for sand tests 1½ pounds of sand, thoroughly mixed with ½ pound of cement. The amount of water for neat briquettes may generally be taken at 6½ ounces, and 3 ounces for the sand mixture. The sand may either be a local or a standard sand, but if the local sand is to be used, several careful tests should be made to obtain the correction factor to reduce the results to standard quartz, a few pounds of which should be kept on hand if possible. The mixing and moulding of the briquettes is performed exactly in the manner described in Chapter IX.

For twenty-four hours after making, the briquettes must be kept in a damp atmosphere. If no suitable box can be obtained for a make-shift closet, the briquettes must be covered with a damp cloth, the points to be remembered being that the cloth must entirely cover even the sides of the moulds so that no dry air can penetrate under it, that the cloth must be kept wet by allowing the ends to rest in water, and that it must not touch the briquettes. A simple closet may be made by utilizing an ordinary wash tub. Two or three inches of water are poured in; a shelf made of two bricks and a short length of board; and a cover made from a piece of old blanket held on by a few tacks, the ends hanging inside of the tub and touching the water. Any such appliance should be kept in a cool and, if possible, damp place, never exposed to sunlight or a current or air.

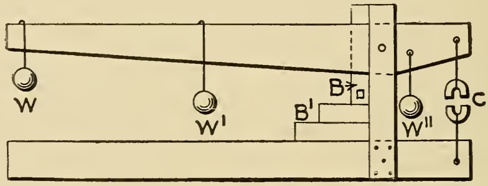


FIG. 120 —Home-Made Cement Testing Machine.

The briquettes may be stored in water in any suitable contrivance, the same tub serving admirably. The briquettes should be placed on their sides, never flat; the water should be fairly pure, neither hot nor cold, and must be changed at least once a week. Pouring in extra water to replace that evaporated will not answer.

For breaking the briquettes a spring balance testing machine, similar to that shown in Fig. 74, is recommended. This machine costs from 60 to 80 dollars and is a good investment for any engineer using much cement, and if many tests are made will soon save its cost in the time and trouble wasted in operating any rough contrivance. If, however, but a few tests are to be made and the magnitude of the work will not warrant such an expenditure, a device like that shown in Fig. 120 may be employed. This particular machine was devised by F. W. Bruce, and was described in Engineering News,* by Lieut. W. M. Black, as follows:

*Vol. XV., page 364.

"The machine consists essentially of a counterpoised wooden lever 10 ft. long, working on a horizontal pin between two broad uprights 20 ins. from one end. Along the top of the long arm runs a grooved wheel carrying a weight. The distances from the fulcrum in feet and inches are marked on the surface of the lever. A clip for tensile tests is suspended from the short arm, 18 ins. from the fulcrum. Pressure for shearing and compressive stresses is communicated through a loose upright, set under the long arm at any desired distance (generally 6 or 12 ins.) from the fulcrum. The lower clip for tensile strains is fastened to the bed-plate. On this plate the cube to be crushed rests between blocks of wood, and to it is fastened an upright with a square mortise at the proper height for

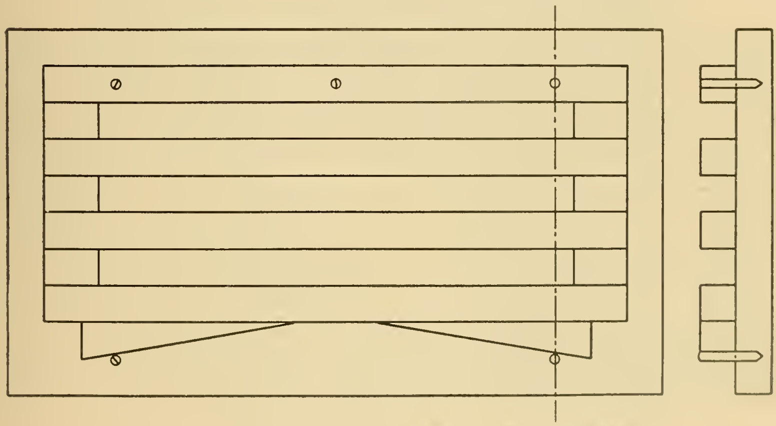


FIG. 121.—A Simple Mould for Making Prisms of Mortar.

blocks to be sheared. The rail on which the wheel runs is a piece of light T-iron fastened on top of the lever. The pin is iron, and the pin-holes are reinforced by iron washers. The clamps are wood, and are fastened by clevis joints to the lever arm and bed-plate respectively. When great stresses are desired, extra weights are hung on the end of the long arm. Pressures of 3,000 pounds have been developed with it."

Another more elaborate home-made cement testing machine will be found described in Engineering News, Vol. XV., page 310. Such an apparatus, however, will cost almost as much as one of the spring balance machines, is much more difficult to operate, and gives far less satisfactory results. Generally, therefore, it is advisable either to procure a small regular cement testing machine if the amount of testing will warrant it, and if not to make tests of strength by cross-breaking, for which the apparatus is much simpler and more easily operated, and the results fully as accurate, if not more so, than those obtained in tensile tests with a crude device.

Strength—By the Transverse Test.—This test is made on rectangular prisms—1 × 1 × 6 ins., or 1 × 1 × 12 ins., preferably the latter. As all tests for strength are better criteria of the quality of the material when made of sand mortar than when of neat cement, the sand tests should be those generally made. As with tensile briquettes, it is preferable to make them of standard sand, but a local sand may be used if the correction factor is

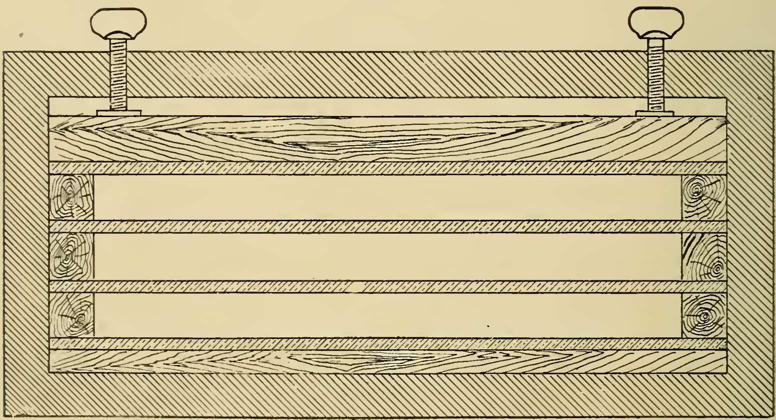


FIG. 122.—A More Elaborate Prism Mould.

first obtained. Sand-mortar prisms have the advantage of being made with greater uniformity than those of neat cement, and may be tested with the simplest sort of device.

The same general method for the making and storing of these prisms, already described for briquettes, is followed. A mould may be made of planed one-inch boards, and some inch wide strips of iron, or entirely of wood as shown in Fig. 121. A convenient mould of cast-iron is shown in Fig. 122, which will cost but 2 or 3 dollars, and will last for many tests. Any mould should be well oiled before use.

For breaking the mortar prisms the simple arrangement shown in Fig. 123 is all that is necessary. The knife edges are made from a round piece of wood, one inch in diameter, and the load applied by pouring sand into a bucket. For testing short prisms, heavier prisms or those of neat cement, a stronger arrangement for carrying the load must be provided, or some other device employed, but generally this easily made arrangement will be entirely sufficient. A more elaborate machine for transverse tests is described in Engineering News, Vol. XXX., page 469, the load being applied by pouring water into a pail, which operates through a long lever. A lever machine like that in Fig. 120, may also be made if desired. The author believes, however, in either purchasing a tensile machine or else in mak-

FIG. 123.—Illustrating the Method of Making Transverse Tests Without Apparatus.

ing rough transverse tests as described, for with the cheap tensile machines now in the market it is difficult to imagine a case in which a piece of work was too small to warrant such a purchase, but which would require the employment of an elaborate but tedious and inaccurate home-made device.

The results of cross-breaking tests are expressed by the formula $\frac{3wl}{2bh^2}$, in which w = the center load, l = the span, b = the width and h = the depth of the specimen. For one-inch rectangles this becomes 3/2 w l. The results of cross-breaking tests expressed by this formula have been shown to give values from $1\frac{1}{4}$ to 3 times the tensile value, depending upon the length, dimensions, richness, age and method of treatment of the specimen. For seven and twenty-eight day tests made on prisms of 1 : 3 sand mortar, one inch square and on a span of 10 inches the factor is very nearly 1.5. If, therefore, a prism 7 or 28 days old be broken on this span the center load will be exactly one-tenth of the tensile strength. The procedure, therefore, is to make prisms 1 × 1 × 12 ins. of 1 : 3 sand mortar and to break them on a span of 10 ins. at 7 and 28 days, the center load causing rupture equaling one-tenth of the tensile strength in both cases. It is safe to assume that the average of 3 prisms will give a tensile value accurate to at least 30 per cent., so that if 170 and 240 pounds are specified for the tensile strength at 7 and at 28 days, tests carrying a center load of less than 12 and 16 pounds at these periods can be considered as doubtful, and those carrying less than 9 pounds at 7 and 12 pounds at 28 days may be rejected with comparative certainty. Although this may not ensure absolutely first-class material, it is far better than nothing and will positively preclude the use of worthless cements. No apparatus, other than a pair of scales, is required except what may readily be made with a few tools.

Table LI. gives a series of tests made by the author to show the accuracy of rough tests made in this manner (Fig. 123). The values of tensile strength are the average of 3 briquettes, while 3 prisms make the average for cross-breaking. It may be observed that the mean error of the prisms between themselves averages but 5.3%, while the average error between briquettes and prisms amounts to but 5.6%. It is believed that this will be found the most satisfactory method for making approximate tests of strength.

Soundness.—The normal pat and boiling tests are recommended for determinations of soundness, and their manner of conduct is exactly similar to that already described in Chapter X. A paste of neat cement of normal consistency is made, and two circular pats about $3\frac{1}{2}$ ins. in diameter, $1\frac{1}{2}$ in. thick at the center and tapering to thin edges, and a round ball $1\frac{1}{4}$ ins. in diameter are formed. Enough paste will be left over from the setting test to make these specimens, so that only one mixing is required for the two tests. The pats should be moulded on plates of glass about 4 ins. square and not less than $\frac{1}{8}$-in. thick.

TABLE LI.—Showing the Accuracy of Transverse Tests Made in the Manner Illustrated in Figure 123.

No.	Kind of Sand (20-30 Size)	7 Days: Ultimate Center Load in Pounds—on Prism 1-in. x 1-in. with Span of 10-in.—Multiplied by 10: 1	2	3	Average	Tensile Strength Pounds per sq. in.	Error of Prism to Tensile Strength: Lbs.	Per cent.	Average Error of Single Prisms: Per cent.	28 Days: Ultimate Center Load in Pounds—on Prism 1-in. x 1-in. with Span of 10-in.—Multiplied by 10: 1	2	3	Average	Tensile Strength Pounds per sq. in.	Error of Prism to Tensile Strength: Lbs.	Per cent.	Average Error of Single Prisms: Per cent.
1	Crushed Quartz	222	250	236	236	236	0	0	3.9	289	305	241	278	301	— 23	7.6	9.0
2	"	210	205	203	206	201	+ 5	2.5	1.3	254	290	277	274	277	— 3	1.1	4.7
3	"	175	180	197	184	167	+ 17	10.2	4.7	279	272	262	271	262	+ 9	3.4	2.2
4	"	225	225	234	228	218	+ 10	4.6	1.8	322	346	219	296	309	— 13	4.2	17.2
5	"	222	240	246	236	269	— 33	12.3	3.9	309	336	355	333	333	0	0	4.9
6	"	240	227	218	228	210	+ 18	8.6	3.4	299	305	322	309	306	+ 3	1.0	2.9
7	"	217	255	270	247	237	+ 10	4.2	8.2	304	339	272	305	294	+ 11	3.7	7.4
8	"	245	245	226	239	237	+ 2	0.8	3.5	262	289	235	262	275	— 13	4.7	6.9
9	"	251	291	240	261	267	— 6	2.2	7.8	351	310	312	324	324	0	0	5.5
10	"	. . .	214	194	204	226	— 22	9.7	4.9	300	329	322	317	306	+ 11	3.6	3.6
11	"	222	250	232	235	218	+ 17	7.8	4.4	335	337	312	328	305	+ 23	7.5	3.3
12	"	271	226	221	239	225	+ 14	6.2	8.8	297	302	304	301	303	— 2	0.7	0.9
13	"	194	222	202	206	200	+ 6	3.0	5.2	. . .	259	267	263	260	+ 3	1.2	1.5
14	"	298	241	224	254	255	— 1	0.4	11.4	336	290	297	308	333	— 25	7.5	6.2
15	"	258	262	213	244	255	— 11	4.3	8.6	291	251	282	275	254	+ 21	8.3	5.7
16	"	211	214	195	207	210	— 3	1.4	3.7	253	259	245	252	273	— 21	7.7	2.0
17	"	242	248	220	237	255	— 18	7.1	4.6	312	271	312	298	284	+ 14	4.9	6.2
18	Ottawa	365	410	394	390	344	+ 46	13.4	4.2	452	521	480	484	488	— 4	0.8	5.0
19	"	345	337	330	337	323	+ 14	4.3	1.5	462	391	394	416	438	— 22	5.0	7.5
20	"	286	376	367	343	361	— 18	5.0	11.1	381	352	277	337	342	— 5	1.5	11.8
21	"	220	266	222	236	259	— 23	8.9	8.5	384	384	269	346	338	+ 8	2.4	14.7
22	"	377	377	298	351	320	+ 31	9.7	10.0	418	393	373	395	390	+ 5	1.3	4.0
23	"	238	269	248	252	256	— 4	1.6	4.6	396	357	373	375	361	+ 14	3.9	3.6
24	Bar	191	210	201	201	199	+ 2	1.0	3.2	222	243	193	219	245	— 26	10.6	8.1
25	"	167	168	157	164	185	— 21	11.4	2.8	259	255	262	259	252	+ 7	2.8	0.9
26	"	145	140	. . .	143	150	— 7	4.7	1.7	182	201	. . .	192	219	— 27	12.3	4.9
27	"	148	137	143	143	129	+ 14	10.9	2.6	260	233	235	243	206	+ 37	18.0	4.8
28	"	191	156	145	164	169	— 5	3.0	11.0	285	318	260	288	297	— 9	3.0	7.1
29	"	183	169	198	183	165	+ 18	10.9	5.3	230	251	231	237	222	+ 15	6.8	3.8

Notes—All tests on mortars of 1 part Portland cement to 3 parts sand. Tensile strength based on 3 briquettes only.

The specimens, like briquettes and prisms, are kept in damp air for 24 hours, after which one of the pats is placed in water with the strength test-pieces, and the other kept in air protected from sunlight or excessive heat. These pats may be examined as often as desired, but should be kept for at least 28 days. Their condition is examined in accordance with the methods outlined on page 163.

The ball for the boiling test, after having been kept 24 hours in moist air, is placed in clear water at normal temperature and gradually heated so that the water is brought to a boil in about half an hour, and kept in boiling water for 3 hours, after which it is removed and examined for checking or cracking. Any pail or can may be employed for making the test, the only points to be considered being that the volume of water should be not less than a quart, and that some arrangement such as a bent piece of wire netting should be placed in its bottom to prevent the specimen from touching it. A second pail of water should be kept boiling beside the first, from which water is poured from time to time to replace that evaporated: the addition of cold water may chill the specimen and affect the results. Illustrations of samples passing and failing in this test are shown in Figs. 80 and 81. Cements failing in the normal pat tests should invariably be rejected. For the interpretation of the boiling test, see pages 175 to 182.

Purity.*—For this test, provide a 4 ounce bottle of dilute (1 : 1) hydrochloric acid, a ½ ounce bottle of acetate of lead and a deep china or glass saucer. Place about as much cement as can be lifted on a quarter-dollar coin in the saucer and pour on it enough acid to cover it, stirring at the same time.

Pure Portland cement effervesces violently for a second or two, leaving a residue of reddish jelly, which on the addition of more water goes entirely into solution except for a few flakes of silica. Adulterations of limestone, lime and natural cement effervesce much longer and generally leaves a residue of black gritty particles, which can be examined by adding more water to the mixture, the cement going entirely into solution except for a few floating flakes of pure white silica, while the residue remains. Adulterations with cinder, sand, slag, or similar materials also leave the gritty residue. The presence of slag may

*These as well as other tests for adulteration are described more fully in Chapter XI., page 204.

be detected by the odor of hydrogen sulphide on the addition of the acid or by placing a strip of newspaper moistened in lead acetate over the saucer, which turns black or brown if slag is present.

Apparatus.—The apparatus employed in the making of these tests, determining strength in cross-breaking, is as follows:

Postal scale—4 ounces—$\frac{1}{4}$ ounce.
Pan scale—30 pounds—$\frac{1}{4}$ pound.
Sieve—100 mesh.
Glass graduate—8 fluid ounces capacity.
Linen-tester's microscope—$\frac{1}{2}$-in. opening.
Small box of standard quartz sand (25 pounds).
Six-inch pointing trowel.
Six glass plates (4×4 ins.)
Hydrochloric acid (1 : 1 dilute)—4 ounce bottle.
Acetate of lead—$\frac{1}{2}$ ounce bottle.

The other devices employed are such as may be obtained in any place, or which may be made with a few carpenter's tools. The cost of the articles in this list should not exceed ten dollars. For making the tensile test a small machine and a few moulds must be purchased, which will cost from 70 to 90 dollars, but which, for any one using much cement, is money well expended. Most engineers will already have all or part of the articles listed, so that the actual cost of apparatus is practically nil and at the same time the results of the tests give a very fair indication of the quality of the material and will always preclude the use of worthless or dangerous cements.

Interpretation of Results.—The general interpretation of results has already been considered at some length so will not be repeated here, but it is advised that the standard methods of conducting the tests as well as the conditions affecting them and the consideration of results be studied carefully before attempting to employ these cruder methods, so that just what reliance to place on them may be clearly understood. It would also be well, before making tests for the purpose of accepting or rejecting a shipment, to examine some cements known to be good, and if possible, one of inferior quality, to obtain practice in the conduct of the determinations. An unsound cement may be prepared by adding from 5 to 10 per cent. of ground un-slaked lime to a normal cement.

It is always safe to reject cement failing in the normal pat tests, and generally if failing in strength (less than 135 lbs. modulus at 7 days) or showing adulteration. Cement failing in boiling, fineness, setting, or giving a transverse modulus of less than 180 pounds at 7 days tested in a 1 : 3 mortar, should be regarded as suspicious and inferior to one passing these tests.

CHAPTER XIV.

PRACTICAL OPERATION.

Equipment.—It is difficult if not impossible to give a list of apparatus for cement testing, that would entirely satisfy the needs of any particular laboratory and yet not include much unnecessary equipment. Each case therefore requires individual treatment according to the nature of the work and the demands upon the laboratory. The following list, however, is an attempt to give all the apparatus required by a large, fully-equipped permanent laboratory, or a field laboratory connected with construction of unusual importance. The outfit required for the testing connected with a mill, or with a fairly large piece of construction work may be obtained by omitting from the list those articles preceded with a star (*). The quantities given are based upon a maximum testing capacity of eight samples a day, so that a greater or less estimated capacity will require alteration in the number of those articles marked with a dagger (†).

- 1 "shot" cement testing machine (capacity 1,000 lbs.).
- *1 "long lever" cement testing machine, with attachments for compressive and transverse tests (capacity 2,000 lbs.).
- *1 "universal" testing machine (capacity 150,000 lbs.).
- 1 scales for cement (1,500 grams—1 gram).
- 1 scales for fineness (100 grams—0.1 gram).
- *1 scales for specific gravity (100 grams—0.01 gram).
- 1 pan scales for rough weighing (50 pounds—¼ ounce).
- †16 4 gang briquette moulds.
- †16 3 gang briquette moulds.
- *†4 iron moulds for 6-inch cubes.
- *†4 moulds for 2-inch cubes.
- *†4 moulds for 1-inch cubes.
- *†4 moulds for prisms (1 x 1 x 13 inches).
- *†2 moulds for prisms (2 x 2 x 13 inches).
- 1 Vicat needle, with plunger.
- †8 additional rubber rings for Vicat needle.
- †4 No. 200 sieves for testing fineness.
- †2 No. 100 sieves for testing fineness.
- 1 No. 50 sieve for testing fineness.
- 1 each of No. 20 and No. 30 sieves for testing standard sand.
- *1 each of No. 10, No. 40, No. 74 and No. 150 sieves for sand testing.
- *1 each of sieves of openings of ¼″, ½″, ¾″ and 1″ for stone testing.
- †2 "Le Chatelier" specific gravity bottles.
- 1 6-inch funnel and stand.
- 1 precipitating jar.
- 2 each of No. 4 and No. 10 beakers.
- 2 graduates—150 c. c.
- 2 graduates—350 c. c.
- *1 graduated cylinder—1,000 c.c.
- *1 cubic foot measure.
- †2 chemical thermometers—100° C.

- 1 room thermometer.
- 1 steaming and boiling apparatus for soundness tests.
- 1 mixing table, or glass mixing slab (24 x 24 x 7/8 inches).
- Storage tanks or pans for briquettes.
- 1 damp closet.
- †250 glass plates—4" x 4" x 1/8".
- †8 glass strips (3" x ¾" x length of damp closet).
- 1 doz.—6" pointing trowels.
- 1 3" pointing trowel.
- 6 pairs rubber gloves (with gauntlets).
- 1 doz. sink scrub brushes.
- ½ gross—marking pencils.
- †1 barrel standard sand.
- *2 Bunsen burners.
- †200 sample cans.
- †3 collecting cans.
- 1 sampling auger.
- 4 6-inch scoops.
- 1 sand glass (1 or 1½ min.).
- 1 clock.
- 1 bag No. 10 shot (25 pounds).
- †3 galvanized iron waste cans.
- 1 oil can, and motor oil.
- 6 brushes (assorted sizes).
- Box of carpenter's tools.
- Glass rods and tubing.
- 1 each linen tester's microscopes (½" and ¼" opening)
- *1 microscope (1-inch objective).
- 1 4" evaporating dish.
- Bottle hydrochloric acid (6 oz.).
- Bottle acetate of lead (1 oz.).
- Bottle methylene iodide (2 oz.).
- Bottle benzole (4 oz.).
- †Can benzine (2 gallons).
- Separatory funnel for testing adulterations.

In addition there must be provided tables, shelves, etc., as well as connections for gas, water and light. If a universal testing machine is installed, provision must also be made for power, preferably from an electric motor. A small fan motor should also supply power for operating the long lever cement testing machine. If it is desired to install an equipment for making chemical analyses, the list of apparatus required will be found on page 207.

A simple equipment for a small field laboratory, testing not more than 2 or 3 samples a day, and only on specification requirements is contained in the following list:

Cement testing machine (shot or spring balance).
Pair scales for weighing cement.
Pair scales for fineness.
Briquette moulds.
Mixing plate of glass 24" × 24" × ¾".
Vicat or Gillmore needles.
Sieves for fineness—No. 100 and No. 200.
Sieves for sand—No. 20 and No. 30.
Le Chatelier specific gravity bottle.
Can of benzine.
Glass graduate (200 c. c.)
Thermometer.
Boiling apparatus.
Storage pans.

Damp closet or arrangement for damp cloth.
Glass plates ($4'' \times 4'' \times \frac{1}{8}''$).
Glass strips ($3''$ wide—$\frac{3}{4}''$ thick and suitable length).
$6''$ trowel.
Rubber gloves.
Standard sand.
Sample cans.

For making occasional or approximate tests, an extremely simple outfit has already been described on page 237. The four classes of equipment given represent the average of most testing laboratories, but every particular instance is so markedly different, that the necessary equipment is a very variable quantity, so that each engineer must decide what is needed to meet his own individual conditions. The different lists given, however, may possibly serve as guides in this selection.

Force.—To operate properly even the smallest of field laboratories, the testing should be performed under the direct personal supervision of a technically educated man, for it is only the man trained to making scientific observations who fully appreciates the importance of standardizing details and following them closely. It is seldom that even the best practical operators, when pushed for time, will not shorten the time of mixing or increase the rate of breaking briquettes, unless he knows that he will be called to account for so doing. It is not that the man is not conscientious in his work, but that he fails to appreciate the amount of error introduced by seemingly trivial deviations from the fixed method. The technical man, on the other hand, fully understands these conditions, and his only excuse for permitting work of this character is wilful neglect or carelessness.

The number of men required to operate a laboratory, making the ordinary specification tests, will average one man in charge, and one helper for every four samples per day on the estimated capacity. Thus two men can readily test four samples of cement a day; three men, eight samples, and so on. This, however, implies well trained and experienced operators; green men will be fortunate to accomplish half this amount of testing. If it is desired to make chemical analyses, another technically educated man must be added to the force. Also, if the daily amount of testing averages over 12 samples, it may be necessary to employ a clerk for recording and reporting the results.

The salary of the man in charge will be from 60 dollars per month up, depending upon the size and importance of the laboratory and upon the amount of responsibility placed upon him. The salaries of the helpers will range from 30 to 75 dollars per month, while the services of a chemist will cost from 60 to 100 dollars.

The average field laboratory connected with construction work can be operated by two or three men, at a salary charge of from 100 to 200 dollars a month. A mill laboratory at a plant of about 1,000 barrels daily capacity making both chemical and physical tests can be operated by a chemist in charge and 2 or 3 helpers, at a salary cost of about 200 to 300 dollars a month.

Cost of Operation.—The cost of operation of a testing laboratory is, of course, extremely variable, depending upon the number of samples tested, the number of tests made, the amount of experimental work performed in addition to ordinary routine and many other conditions, and hence may range anywhere from $1.50 to $10.00 per sample. Under the most favorable conditions a laboratory may reach the minimum figure given, but such cases must be infrequent. The cost of testing cement in the Philadelphia Laboratories, including salaries, supplies and repairs, but not including heat, light, power, rent or interest on money invested, averages from $2.00 to $2.50 a sample.

The cost of a field laboratory on construction work will average from $3.00 to $5.00 a sample, roughly equivalent to about 3 cents a barrel of cement, or from 3 to 5 cents per yard of concrete, although it may average considerably in excess of this figure under unfavorable conditions. Even at a cost of 5 or 6 cents a yard of concrete, the maintenance of a testing laboratory is tantamount to an insurance of the structure at a remarkably low rate.

The cost of the original equipment will be anywhere from ten to ten thousand dollars in accordance with the amount and character of the apparatus. That employed in the author's laboratory would amount to considerably in excess of the maximum figure. The list given on page 240 as a suitable equipment for a field laboratory should cost about $250, while the first complete list on page 239 would cost from $1,800 to $2,500.

A good outfit for a mill or large field laboratory could be purchased for about $350, or about $600 including apparatus for chemical analysis. Both the cost of equipment and maintenance are therefore seen to vary between wide limits, depending upon the particular conditions to be met, but the foregoing will serve as a rough guide for the estimation of cost in any individual instance.

Operation.—Probably the most important factor in the efficient operation of a cement testing laboratory is in the employment of experienced and conscientious men. No matter how carefully a standard method of manipulation be followed, an inexperienced operator will at first obtain most inaccurate results, and the only way to train him properly is to require him to work day after day beside an experienced man and from the differences in the results learn where to locate his errors. Most of the operations of cement testing may be learned after practice of a week or two, but the proper making of briquettes cannot be acquired in less than a month's hard work under intelligent supervision. If it is necessary to employ a green man, on first organizing a laboratory, the results of his early tests should be interpreted with a large provision for error. The uniformity of his work may be tested by computation of the probable or average error of his results, but systematic errors can only be detected by most careful supervision and by comparison of the values obtained with those of a well-established laboratory. Where the force consists of several men, it is also advisable to have trained under-studies for each man's work, so that in case of any one man's sickness or retirement another can take his place without the confusion attending the breaking in of a new man.

Another advisable procedure is to have printed or typewritten copies of the standard methods of testing in the hands of each operator, and to make certain that he is entirely familiar with all the steps of the process, and understands the importance of adhering to every detail. A wilful violation or departure from the fixed methods to gain time or to make the work easier should result in his instant dismissal from the force. The difficulty of instilling the importance of detail in the minds of practical but uneducated men makes imperative the constant supervision of a man of scientific training, for otherwise the character of the work soon becomes so slipshod that the results of the tests are almost valueless.

In formulating a standard method of testing, the great difficulty lies in determining the line where economy of time and labor at the expense of accuracy must stop, the only proper method being first to learn what accuracy is essential to the correct interpretation of each test, and then to ascertain whether the probable error or best the maximum error of the methods lies within that limit of accuracy. Thus, in the author's laboratory it is the practice to remove briquettes from the damp closet after 21 instead of 24 hours, it first having been learned that this departure from standard practice had no appreciable influence on the results, while at the same time it much simplified the day's routine. The time of mixing briquettes also has been shortened, to economize labor, to one instead of 1½ minutes, the uniformity and accuracy thus obtained being still well within the allowable limit of error, but mixing for only half a minute was at the same time found to create errors of unjustifiable amount. Thus each step in the process should be considered and the best method adopted.

Another important detail necessary to obtain accuracy, is the systematic recording on regular forms of the results of the tests, made immediately after each determination. Many operators jot down their figures on a slip of paper or the back of an envelope, and then copy them after all the tests are finished, which is likely to cause the making of mistakes, and often creates a temptation to slightly alter the figures if the values are obviously abnormal or show much error. The author once saw an operator in a well-known laboratory break briquettes at values of say 238, 300, 254, 288, and then record say 264, 275, 270, 271, saying "it is just the same thing, and it looks better, you know." This tendency of operators to make their reports look better is largely overcome by requiring each value to be entered on a printed form immediately after each result is obtained.

Three of the laboratory sheets used by the author are shown in Figs. 124, 125 and 126, these being for fineness, setting and briquette reports. Similar sheets are used for pat tests, boiling tests, specific gravity and the other determinations.

Recording Systems.—Different methods of recording and reporting the results are of course necessary to meet different

TESTING LABORATORY

FINENESS TESTS

10 – 5 – 05

No.	50	100	200
54273	0.0	7.3	24.8
74	0.0	6.8	23.4
75	0.2	8.4	26.4
76	0.0	6.6	24.6
77	0.0	6.6	24.0
78	0.0	8.0	24.8
79	0.0	9.0	24.4
80	0.1	6.8	23.6
81	0.0	7.2	24.0
82	0.0	5.8	22.6
83	0.0	6.0	23.0
84	0.0	7.8	23.8
85	0.0	4.8	20.6
54286	0.0	6.6	24.0

W. M.

FIG. 124.—For Fineness Tests.

TESTING LABORATORY.

CEMENT BRIQUETTES.

10 – 5 – 05

Age	No.	1	2	Av'g
28 d.	53929	867	863	865
		307	310	309
	30	754	740	747
		286	291	289
	31	753	762	758
		300	294	297
	32	659	639	649
		235	241	}
		Extra —	243	} 240
	33	804	781	793
		314	315	315
	53934	752	764	758
		275	271	273

C. H. C.

FIG. 126.—For Cement Briquettes.

TESTING LABORATORY

SET TEST

10 – 3 – 05

No.	Brand	% Water	Time of Setting			Paste Temperature		Room Temperature	
			Begin	Init.	Hard.				
54269	A —	19.0	9-30	11-05	3-20	21	24	22	24
70	B —	19.0	9-35	11-45	3-10	21	24	22	24
71	C —	20.0	9-40	10-09	11-05	21	29	22	24
72	D —	20.5	9-44	10-55	2-40	21	27	22	25
73	E —	19.0	9-49	12-05	4-00	21	23	22	23
74	F —	19.5	9-54	11-30	4-10	21	25	22	23
75	G —	21.0	9-59	11-00	3-05	21	26	22	24
76	H —	19.5	10-03	10-55	3-00	21	25	22	24

C. H. C.

FIG. 125.—For Tests of Time of Setting.

LABORATORY REPORT SHEETS.

conditions, but it is thought that a description of those used in the Philadelphia Laboratories, where a large variety of materials are tested for use in many classes of construction, may be of interest. After experimenting with several systems of recording, including record books, loose-leaf books and card-systems, it was found that the last method was far more convenient than any other. A mill laboratory, or one in the field where only one brand of cement is tested for but one piece of construction, may use ledgers to advantage, while where several brands are used the loose-leaf books may be employed, but for the average laboratory the card system will be found the most convenient and satisfactory in the long run. They may be indexed in any way, may be referred to with great ease, while for obtaining average results or for examining the properties of any particular brand among those tested, the amount of time and labor saved is very marked.

The following is a description of the methods employed in the Philadelphia Laboratories for recording and reporting results: Whenever a shipment of cement is received upon any municipal work, the field inspector sends to the laboratory a postal card, on which is stated the size of the shipment, the brand of the cement, the place received and the character of the work for which it is intended. On receipt of this notification a collector is sent to the work, who not only takes a sample of the material in accordance with the methods given in Chapter V., but also examines the shipment as a whole in regard to its condition, the soundness of the packages and its storage, a report of which is submitted with the sample. These samples are brought to the office, placed in sample cans, which are marked with a consecutive number and the notification card given the same number and filed. The sample is henceforth, during the progress of the tests, known only by its number, so that, even if it were so desired, it would be impossible for any operator to show favoritism to any sample or brand in the conduct of the determinations, the author making a point of marking the samples personally.

After the samples for each day's testing are marked, a distribution sheet (Fig. 127) is made out, giving the number of briquettes to be made from each sample and the ages at which they are to be broken, it being assumed that 7 and 28 day tests

TESTING LABORATORY

DISTRIBUTION SHEET

10 – 5 – 05

No.	Age	Remarks
54273	2 m.	
74	4 m.	
75	6 m.	
76	1 year	
77	2-4-6 m.-1 yr.	14 n - 12
78	3 m.	
79	3 years	
80	2-6 m. 1 yr.	12 n - 10 s
81	2 m.	
82	——	7 and 28 only
83	4 m.	
84	6 m.	Sand 1:2, 1:3, & 1:5
85	3 years	
54286	5 years	

E. M. F.

FIG. 127.—Distribution Sheet.

TESTING LABORATORY

BREAKING SHEET

10 – 3 – 05

Age	Briquettes
7 Days	54130 to 54144
28 Days	53912 to 53928
2 Mos.	53731
3 Mos.	53429-34-35-42
4 Mos.	
6 Mos.	52936-41
1 Year	50997-98-99
2 Years	47986
3 Years	
5 Years	42761-65-82
7 Years	
10 Years	
7 days.	Specials - O.E. - 12 & 13.

FIG. 128.—Breaking Sheet.

NUMBER		BRAND					DATE TESTED	
52937		——					1/4/05	
SHIPMENT		PLACE OF COLLECTION					DATE COLLECTED	
600 bags		12th & Market Sts.					1/3	
FINENESS	50: 0.0	100: 7.8	200: 25.0	SPECIFIC GRAVITY			3.184	
SETTING	INITIAL: 108	HARD: 408	RISE: 3	TEMPERATURE: 21 - 21			% WATER: 20.5	
STRENGTH	1D.	177	7D.	738	28D	823	2	856
	1 : 3 STA. SAND		7D.	215	28D.	304	m.	329
PATS	AIR-7D.: Lg.	AIR-28D.: Lg.	WATER-7D.: O.K.	WATER-28D.: V.S.C. Lg.				

REMARKS:— Car - P.&R. #72309. 2 m. - P.N.C.

Boiling test - OK.

% - SO_3 - 1.67.

FIG. 129.—Card Record Used in the Philadelphia Laboratories.

are always made, while, if not otherwise stated, the number of briquettes is taken to be eight neat and six sand. At the same time, the briquettes to be broken at each period are entered in diaries arranged 10 years ahead, so that they show for each date exactly what tests are then to be made. For example, if briquettes are put up on May 1, 1905, to be broken at 7 and 28 days, 3 months and 1 year, the sample number is entered under May 8th, May 29th, August 1st, 1905, and May 1st, 1906.

After the briquettes have been made, and on the following day removed from the moulds and marked, they are placed in the storage tanks, which are divided by sections into periods of 1, 7 and 28 days, 2, 3, 4 and 6 months, and 1, 2, 3, 5, 7 and 10 years. With the distribution sheet as a guide, each briquette is placed in the section marked with the age when the test is due, those in each section being placed in the order of making, so that the beginning of the first row contains the first briquettes to be broken. Each day a breaking sheet (Fig. 128) is copied from the diary giving the tests due on that date; with this sheet, the operator breaking the briquettes can go to each section of the tanks and remove the first briquettes in the different rows, whose numbers should correspond with those on the sheet, thus avoiding any waste of time in hunting for the different briquettes, insuring that every test is made on the proper date since by this method it is impossible to overlook them, and also requiring a minimum storage capacity in the tanks. Of course, for handling a small number of briquettes, so elaborate a system would be unnecessary, but when the briquettes in storage run well into the tens and twenties of thousands, the lack of such system means much wasted time and energy. No matter how few tests are made, the use of diaries, in which, under each date, are entered the tests when due, will be found most convenient.

When any determination is made, the results are at once entered on blanks similar to those shown in Figs. 124 to 126, eight different forms being employed in routine cement testing, all of which are sent to the laboratory office twice a day for entry. The permanent records consist of a consecutive number book and a file of card records. In the consecutive number book are entered, when each sample is received, its number, the size of shipment, the number of the car (if in car load lots), the place and nature of the work where it is intended for use, the date received, the date tested, the brand of the cement, the number of briquettes made and the periods when the tests are due. From this data the first two lines of the card (Fig. 129) are made out, while the other spaces are filled from the labor-

(OFFICIAL HEADING)

The following are results of tests of samples of cement intended for use by the Bureau of .... Division.

Place of Collection,				
Brand,				
Number of Car,				
Specific Gravity,				
FINENESS:				
% Residue on No. 50 Sieve,				
" " " " *100* "				
" " " " *200* "				
TIME OF SETTING:				
Initial Set (in minutes),				
Hard Set " "				
TENSILE STRENGTH:				
24 hours, neat,				
7 days, "				
28 days, "				
7 days, parts sta. sand,				
28 days, parts sta. sand,				
Boiling Test,				

Remarks:

Report approved, *Respectfully submitted,*

Chief Engineer. *Engineer of Tests.*

Fig. 130.—Report of Cement Tests.

atory sheets as they are reported, the sheets themselves being filed for possible future reference. Only the average strength is recorded on the card, while a special notation is employed for the pat tests, V. S. C. Lg. standing for very slightly curled, left glass, etc. The cards for natural cement are similar, except that the strength is tested with 2 instead of 3 parts of sand, and

TESTING LABORATORY

Sheet No. ______ Brand ____________ Year ______

Sample	[illegible]	Fineness			Specific	Setting		Temperature		%	Tensile Strength—Neat							Tensile Strength—Sand					
Number		50	100	200	Gravity	Initial	Hard	Begin	End	Water	1	7	28	2	3	4	6	7	28	2	3	4	6
Total																							
Number																							

Fig. 131.—Balance Sheet.

are of buff color, while the Portland cards are white. They are filed in rotation until the 28 day tests are made, after which they are cross-indexed and filed by brands and by the periods when the "long-time" tests are due.

Reports as shown in Fig. 130 are made at the expiration of 7 and 28 days, any values failing to meet the requirements being stamped with a red star. As an aid to averaging the results for the annual report, and also to enable one to examine rapidly the run of any particular brand, it has been found convenient to employ balance sheets (Fig. 131), on which the values are entered after the 28 day tests have been recorded, and the total value of each column, with the number of tests comprising it, completed on each sheet; with these sheets it is possible at any time to obtain the average of the results of any brand within a few minutes.

The records of experimental tests and investigations, as well as of special tests not in regular routine, are kept in ordinary record books, the subjects being indexed on the card system.

This system has been evolved after the experience of almost 15 years in handling a great variety of many materials under varying conditions, and has been proven the most convenient and satisfactory of the many systems tried.

CHAPTER XV.

NATURAL, SLAG AND OTHER CEMENTS.

NATURAL CEMENTS.

Natural cements are those made by the burning and subsequent pulverization of an argillaceous or argillo-magnesian limestone, and, as the name implies, are made from a single variety of natural rock, without previous pulverization or blending with other materials. In burning the heat is never carried to clinkering temperature.

These cements are made from materials of widely variant character and composition, so that the characteristics of the different varieties bear little similarity to each other. The two best known groups of this material are the "Rosendale" and "Louisville," both of which, however, include several brands of more or less varying composition. These group names are often incorrectly used to cover the entire class of natural cements.

Production.—Natural cement was first produced in the United States in the year 1818, six years prior to the date of Aspdin's patent on Portland cement and 57 years before it was made in this country. The first works were near Fayetteville, N. Y., and large quantities of the product were used in the construction of the Erie Canal. The growth of the industry may be seen by reference to Table I., page 4. It will be observed that for the past 12 to 15 years the production has remained practically constant, this being due to the great increase in the popularity and use of Portland cement. The production by states or districts is shown in Table LII., the New York or "Rosendale" district, leading in both number of mills and amount produced, with the Indiana-Kentucky or "Louisville" district second.

Manufacture.—As compared with Portland, the manufacture of natural cements is a very simple process. The raw rock is either mined or quarried and conveyed to the works in sizes from a "two-man" stone down. Some of the better mills make a practice of running all the rock through a crusher of the Blake type to secure some uniformity in size, but most burn the stone as quarried, without any preliminary treatment.

The rock is burned in plain vertical cylindrical kilns (Fig. 132) made of stone, brick, or iron, lined with fire brick, and operating continuously. The dimensions of the kilns vary greatly in different localities, depending on the rock, the fuel and various other conditions, but usually are from 30 to 45 feet in height and from 8 to 16 feet in diameter, the lower 5 or 6 feet, tapering to a reduced section. Both anthracite and bituminous coals are employed in the burning. The rock and fuel are fed into the top of the kiln in alternate layers, the amount of fuel averaging from 10 to 20 per cent. of the weight of the rock, while the temperature maintained in the kilns varies from 700° to 1,000° C. The burning is continued until most of the carbonic acid is driven off, and until the rock is porous and friable, and when complete the material is drawn from the bottom.

TABLE LII.—Production of Natural Cement in 1903, by States. (From Mineral Resources, 1903.)

State	Number of Works	Quantity Barrels	Value
Georgia	2	80,620	$44,402
Illinois	3	543,132	178,900
Indiana and Kentucky	15	1,533,573	765,786
Kansas	*a.* 2	226,293	169,155
Maryland	4	269,957	138,619
Minnesota	2	175,000	78,750
New York	20	2,417,137	1,510,529
North Dakota	1	....	....
Ohio	*b.* 2	67,025	46,776
Pennsylvania	7	1,339,090	576,269
Texas	2	....	....
Virginia	2	47,922	25 961
West Virginia	1	....	....
Wisconsin	2	330,522	139,373
Total	65	7,030,271	3,675,520

(*a.*) Includes product of North Dakota and Texas.
(*b.*) " " " West Virginia.

On account of the inability to control the burning exactly, a part of the material is overburned and some underburned, thus requiring sorting; the overburned material is often discarded, while that insufficiently burned is given a second calcination.

The first step in the process of grinding is generally to run the calcined stone through a rough crusher known as a "pot cracker" (Fig. 133), which consists of an iron cone, revolving in a shell of similar shape, both surfaces being provided with corrugations. The process then varies greatly in different mills, in some it is screened, the coarse material returning to a finer cracker or edge-runner mill, others send all the material to the fine grinders. These last mills are usually of the old buhr-stone type, 3 to 5 feet in diameter. Emery mills are also employed and often are more economical. A second screening is made in many plants after these mills, the coarse material returning again to them.

In some works, tube mills are employed for the fine grinding; others have batteries of rolls and Griffin mills, or Kent

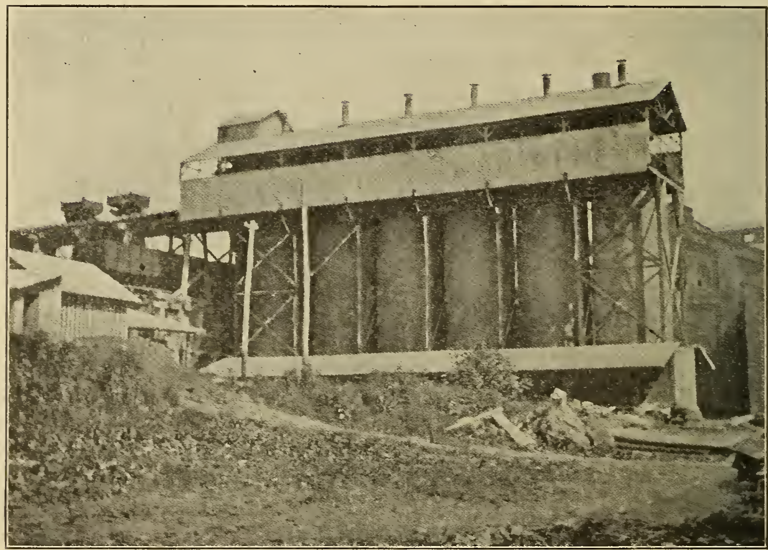


FIG. 132.—Kilns for Burning Natural Cement.

and tube mills. Disintegrators with tube and Griffin mills are also used. The calcined natural rock, on account of the lower heat of burning, is much softer and hence easier to grind than Portland clinker. Figure 134 shows a cross-section through a mill grinding with crackers and buhr-stones, and which is typical of the ordinary natural cement plant.

The finished cement is seldom stored in bulk, but is packed in bags or barrels as soon as it comes from the mills. The net weight of a barrel of natural cement averages 280 pounds, 3 bags being the equivalent of a barrel.

The chief difficulty in the manufacture of natural cement lies in the variations due to the differences in composition between the different strata of rock. This is controlled to some extent by blending in the quarry, and again, in many mills, by a thorough mixing of the finished cement, but even the most thorough precautions will never ensure an absolutely uniform product, and it is this condition that constitutes the chief objection to this grade of material.

Composition.—The composition of a number of varieties of natural cement is shown in Table LIII. It will be observed that

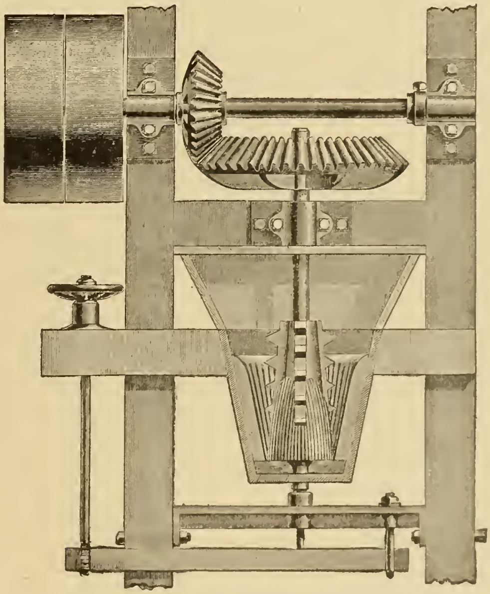


FIG. 133.—Pot Cracker.

the content of silica, magnesia, alkalies and carbonic acid is higher, while the lime is considerably lower than in Portland cement, thus giving a higher hydraulic index. The burning results in the forming of many complex silicates and aluminates, as well as substances of a pozzuolanic character, but unlike Portland this material admits of the formation of many compounds without materially altering its characteristics, whereas all Portland cements are essentially of the same composition. Another lack of similarity between naturals and Portlands is that the content of magnesia in the former acts in a very dif-

TABLE LIII.—Showing the Composition of Commercial Natural Cements from Various Localities.

(From Eckel's "Cement Materials and Industry.")

LOCALITY	Silica $Si\,O_2$	Alumina $Al_2\,O_3$	Iron Oxide $Fe_2\,O_3$	Lime $Ca\,O$	Magnesia $Mg\,O$	Alkalies K_2O+Na_2O	Carbon Dioxide $C\,O_2$	Water H_2O
Utica District—Illinois	27.60	10.60	0.80	33.04	17.26	7.42	2.00	
Louisville District—Kentucky	21.10	7.50		44 40	7.00	0.80	11.18	1.16
" " "	26.40	6.28	1.00	45.22	9.00	4.00	7.86	
Cumberland District—Maryland	28.38	11.71	2.29	43.97	2 21	....	....	
Rosendale District—New York	30.84	7.75	2.11	34 49	17.77	4 00	3.04	
" " " "	22.77	10.43		34.54	21.85	3.63	2.84	1.59
" " " "	27.30	7.14	1.80	35.98	18.00	6 80	2.98	
Central District—New York	20 30	13.67		47.48	18.55	....	....	
Akron-Buffalo District—New York	22.62	7.44	1 40	40.68	22 00	2.23	3.63	
" " " " "	24.30	6.20	2.61	39.45	6 16	5.30	15 23	
Lehigh District—Pennsylvania	18.28	7.43		51.53	2.07	1 50	16.26	
Milwaukee District—Wisconsin	23.16	6.33	1.71	36.08	20.38	5 27	7.07	

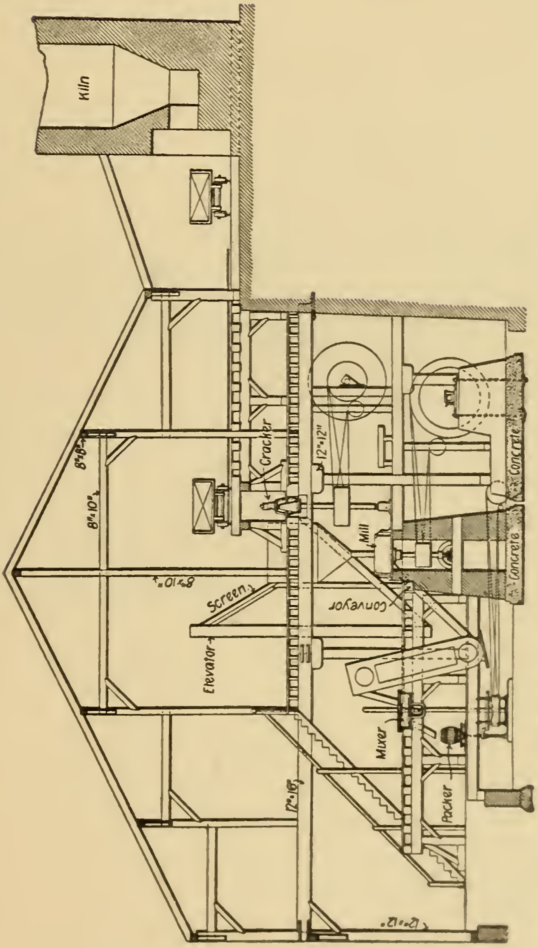


FIG. 134.—Cross-Section of a Natural Cement Plant.

ferent manner and may be present in considerable amount and used in the most unfavorable conditions without injury to the structure. An example of this is in the foundations of the "Brooklyn Bridge" in New York, the masonry foundations of which are laid in a mortar of natural cement containing over 20% magnesia.

Color and Weight.—The color of natural cement is much varied—ranging from a light yellow, to dark gray and even to a chocolate brown. Generally the color is no criterion of quality, except as a measure of the uniformity of a single grade of the material.

On account of the much lower heat in burning, and hence the less complete combination of the ingredients, the weight of natural cements is much less than that of Portlands, being about 45 to 65 pounds per cubic foot against 70 to 90 pounds for Portland, in a loosely filled measure.

Specific Gravity.—The composition, and hence the degree of burning, of natural cements is so variable in character that the specific gravity is generally no criterion of quality regarding the class of material as a whole, but as a measure of the uniformity of a single grade, the test has some slight value. Age and dampness tend to reduce the specific gravity, as with Portlands. The method of conducting the test is similar to that already described, while the results will range from 2.70 to 3.00.

Fineness.—The degree of fineness to which natural cement is ground depends both upon the composition of the material and upon the grinding process employed. Material reduced on buhr-stones will usually have a much larger percentage of very coarse particles than that ground in tube or Griffin mills. The percentage passing a No. 200 sieve averages nearly the same as Portland, although the ratio between the residues on the different sieves is much less constant. For example, the following represents the average of many tests of two brands of natural cement tested in the author's laboratory, the first being reduced on buhr-stones and the second in tube mills:

	Sieve No. 50.	Sieve No. 100.	Sieve No. 200.
No. 1	2.4	12.6	21.8
No. 2	0.1	4.7	21.3

Cement testing less than 15% on the No. 100, and 30% on the No. 200, may be considered acceptable. In general, the effect of fineness on setting and strength, and the method of conducting the determination is similar to that already given for Portland cements. On account of the lower lime content, however, fine grinding is not as essential to soundness with this material.

Time of Setting.—The setting of natural cements is also very variable, but generally is much quicker than Portland, although slow setting naturals are not infrequent. One characteristic of the setting of natural cements is that the ratio of initial to hard set is much greater than with Portlands, the hard set frequently occurring within 15 minutes after an initial set which required 15 or 20 minutes to develop, while a Portland cement acquiring initial set in that time generally requires about 2 or 3 hours, and even longer, to set hard. The effect of age on setting is generally less marked with naturals than with Portlands, the effect of fineness is very similar, while the effect of varying precentages of water is usually more decided with this material.

The normal consistency employed for this test is similar to that for Portlands, but a much greater percentage of water is necessary, ranging from 23 to 35 per cent., depending upon the composition, fineness, age, and other conditions; otherwise the method of testing time of setting is similar to that given in Chapter VIII. Specifications usually stipulate that it shall not acquire initial set in less than 10 minutes, nor hard set in more than 5 hours.

Tensile Strength.—The methods and processes of testing the tensile strength of natural cements are similar to those employed for the testing of Portlands, except that there is far less uniformity of practice in regard to the proportions of the sand mortar specimens, 1 : 1, 1 : 2 and 1 : 3 mixtures all being used by different laboratories. The 1 : 3 mixtures have the objection of being so weak when removed from the moulds that they frequently are spoiled in handling, and also so low in strength at 7 days that it is difficult to obtain accurate results. Mixtures of 1 : 1, on the other hand, are too rich to have the typical properties of sand mortar. For these reasons, it is advisable to test natural cements in a mixture of one part of cement to two parts of sand, this mixture containing enough aggregate to be characteristic of sand strength, and yet strong enough to handle easily, and test accurately at 7 days.

The strength of natural cement is much more regular in its increase with age, and the curves of hardening seldom show retrogression in the strength of either neat or sand tests, in fact any retrogression may be considered as indicative of injurious properties. The effect of age on the cement is to lower gradu-

TABLE LIV.—Effect of Varying Percentages of Water on the Strength of Natural Cement.

(Tests from Paper on the "Tensile Strength of Cement," by E. S. Larned, Proc. Am. Soc. Test. Mats., 1903.)

Brand	Water, per cent.	Sieve test: residue on No. 50	No. 100	No. 180	Wire: minutes Light	Heavy	Tensile Strength 24 hours	7 days	28 days	3 mos.	6 mos.	12 mos.
A......	23	0.1	4.6	10.2	13	32	212	251	252	311	275	356
	25				18	39	185	218	215	289	300	341
	27				21	42	150	188	220	257	272	314
	29				20	52	128	178	202	246	248	256
	31				21	57	112	173	199	224	259	309
	33				27	85	104	172	182	267	246	290
	35				38	137	93	121	178	260	286	319
	37				34	160	85	108	168	262	306	326
	39				67	233	85	119	202	252	371	400
B......	23	2.3	12.4	21.9	22	59	138	177	271	332	284	264
	24				..	78	125	141	264	342	309	310
	25				35	120	150	164	216	308	318	321
	27				49	143	117	116	194	305	345	272
	29				76	166	96	105	164	272	320	267
	31				117	212	72	72	159	270	371	225
	33				115	235	62	71	147	277	379	244
	35				127	400	50	64	112	245	318	315
	37				198	828	59	62	96	...	284	351
	39				260	1057	54	56	85	...	355	364

Note.—Each value is average of six briquettes.

ally both its early and ultimate strength until finally the material possesses little if any cementing properties, although it is always somewhat active as a pozzuolanic material. The effect of the use of different amounts of mixing water is generally greater in natural cements, as may be seen from Table LIV., taken from the same source as Table XXXI. The effect of fineness and of the character of the mixing sand is mechanical in action and, therefore, is similar in both naturals and Portlands. In general, the effect of exterior conditions, is practically similar for both grades of material; the conduct of the test is identical, except for the employment of the richer sand mixture. The normal consistency is obtained with much greater ease by use of the ball method than with the Vicat needle

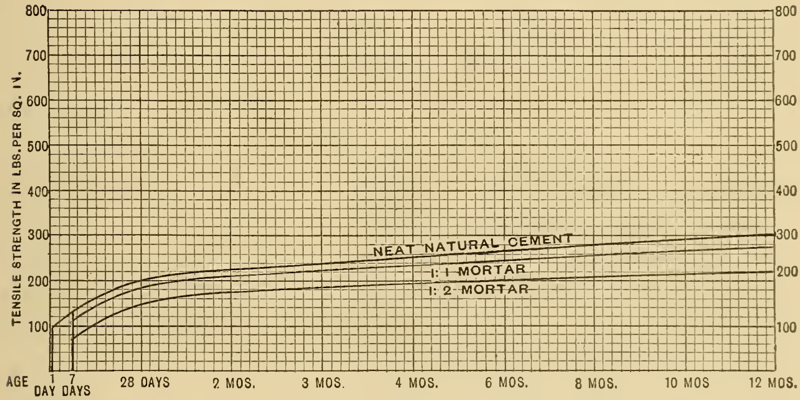


FIG. 135.—The Strength of Natural Cement. (From Taylor and Thompson's "Concrete—Plain and Reinforced.")

for the quick drying out and early setting of the specimen make the latter determination difficult for this material. As with Portlands, the ball method consistency plus one per cent. gives very closely the Vicat needle consistency as recommended by the Committee of the American Society of Civil Engineers.

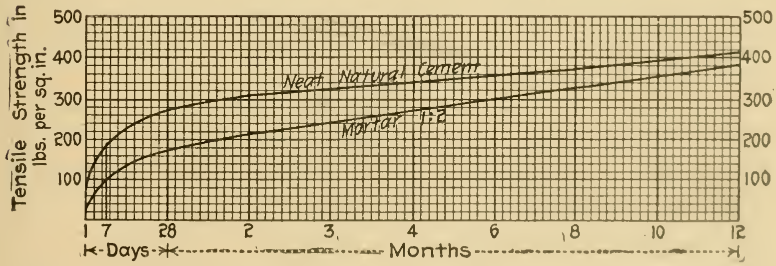


FIG. 136.—The Strength of Natural Cement. (From Tests by the Author.)

gineers. The percentage of water for sand mixtures is given in Table XXXIII., page 111.

The average strength of natural cements is shown in Figs. 135 and 136, and a comparison with the similar curve for Portland cements (Fig 79) shows the rate of hardening to be much more uniform and regular. A good natural cement should develop a strength of 125 pounds at 7, and 225 pounds at 28 days, tested neat, and 75 and 140 pounds at the same periods when tested with two parts of standard quartz sand. When Ottawa sand is employed, the sand strength requirements should be increased 20 or 25 per cent., or to 90 and 170 pounds, respectively. Any retrogression in strength between 7 and 28 days in either neat or sand briquettes is usually indicative of future unsoundness, and such action should always cause the rejection of the shipment. The strength of natural cements is, on account of the less perfect control in manufacture, much more variable than in Portlands, even in different samples of the same brand. Generally those developing the best increase in strength between 7 and 28 days give the highest tests for longer periods, although there are frequent exceptions to this rule. Material passing the above minimum specifications and developing an increase of over 20% between the two periods will, however, be sufficiently strong for all ordinary purposes of construction.

Soundness.—Normal tests, or those made on pats of neat cement paste kept in air and water, are considered to be the only conclusive tests for the soundness of this material. The tests are made, and the specimens examined in accordance with the methods given in Chapter X. Natural cement pats in air seldom adhere to the glass plates, while those in water adhere even more strongly than Portlands. This adherence to the glass plates, however, is no criterion of quality. Excessive expansion, checking, or disintegration in normal pats, is similar in effect with both natural and Portland cements.

Accelerated tests for soundness have frequently been tried, but the consensus of opinion seems to be that their results are misleading and inconclusive. Mr. Sabin* states that comparative tests on briquettes of sand mortar kept in water at 80° C., and at normal temperatures, give a fair indication of the ultimate strengths attained. Tests on pats kept in hot water of a temperature of from 40° to 80° C. have also been advocated, but their employment is infrequent. In general, it is sufficient to employ the normal pat tests alone for natural cement, both on account of the unreliability of the accelerated tests, and also because of the fact that unsoundness as a rule

*In "Cement and Concrete," by L. C. Sabin.

develops much more quickly in naturals than in Portlands, usually within 28 days.

Chemical Analysis.—Except in the mills, analyses of natural cements have practically no value as a guide to their quality, except, possibly, that a measure of the degree of burning may be obtained from the content of carbon dioxide. Methods for chemical analysis, however, have been given in Chapter XI., page 191.

Rough Tests.—For making rough approximate tests of natural cement, as outlined in Chapter XIII., the same methods should be followed except for the omission of the boiling test, and the test for purity.

For the fineness test, 85% should pass the No. 100 sieve. The setting cakes are mixed with from 23 to 35 per cent. of water, as may be necessary to produce normal consistency, and are examined after 10 minutes and 5 hours. Tensile tests are made on neat cement and 1 : 2 mortar, and should develop the different strengths just given. Transverse tests also follow the same methods and are made on both neat and sand specimens, and the results interpreted as for Portlands, allowing at least 25% for errors in the determination and in the reduction factor. Normal pats in air and water are made for soundness tests.

Interpretation of Results.—The strength and the normal pat tests are practically the only criteria of the quality of natural cements, since the fineness of the material has little effect on its soundness or permanency. The fact that unsoundness or failure in strength develops as a rule much earlier in naturals than in Portlands much simplifies the interpretation of results. In general, a natural cement that is sound in the normal pats, passes the given minimum strength requirements, and develops an increase in strength of over 20%, between 7 and 28 days, will be found entirely satisfactory in use. For avoiding difficulties and delays in construction it is well for it to pass the given requirement for setting, although failure in this test, as a rule, but little affects the structural value of the material.

IMPROVED CEMENTS.

Improved cements are of the mixed or blended class, and are made by grinding together natural cement mixed with from 10 to 30 per cent. of Portland clinker.

The methods of testing and the interpretation of results are similar to those employed for naturals, the chief differences between improved and natural cements being the greater strength, the slightly slower set, and the heavier specific gravity. The average results of tests on many samples of improved cements made in the author's laboratory are shown in Fig. 137. The strength specification requirements for improved cements should be increased to 200 and 300 pounds for 7 and 28 days neat, and to 120 and 200 pounds, for 1:2 mortar at the same periods. Ottawa sand mortar should give 145 and 240 pounds.

The advantages of this material are first a more uniform product, on account of the controlling action of the Portland cement, and secondly a much increased strength, which increases more rapidly than the content of Portland cement, or

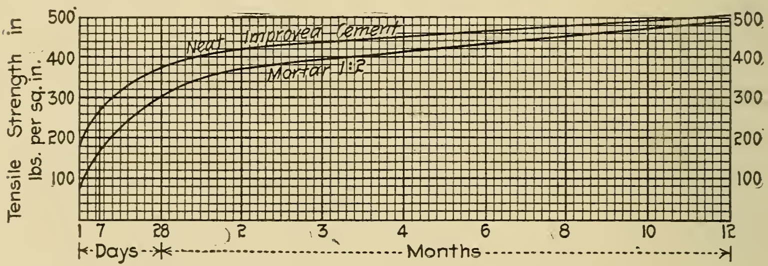


FIG. 137.—The Strength of Improved Cements. (From Tests by the Author.)

the proportionate increase in cost. Results of tests of mixtures of Portland and natural cements are shown in Table LV. Improved cements are especially adapted for masonry and brick work, on account of the fat, rich mortar formed, and the greater strength developed, as compared with those of natural cement.

POZZUOLANA CEMENTS.

Pozzuolana cements (also spelled puzzolana and puzzolan) are hydraulic cements, made by grinding a pozzuolanic material, such as blast furnace slag or volcanic scoria, with dry slaked lime.

A number of natural pozzuolanic materials are employed in Europe for this class of cement, but none have been found in this country. This was the type of cement used by the old Romans in their extensive hydraulic constructions, the pozzuolana being of volcanic origin found near the foot of Mt. Vesuvius at a place called Pozzuoli, from which the material obtained its name. A similar material of volcanic origin known as trass occurs in Germany and Holland, and is employed for the same purpose.

Arenes, a quartzose sand mixed with clay, is another type of pozzuolana found in France. The greater part, however, of pozzuolanic cements, at the present time, are made from an artificial material resulting from the quenching or granulation of blast furnace slag, and since this is the only form of this material made in this country, the others will not be considered further.

SLAG CEMENTS.

Although cement made from a mixture of lime and granulated blast furnace slag has been used in Europe over 20 years,

TABLE LV.—Tensile Strength of Mortars made with Mixtures of Natural and Portland Cement.

(From Sabin's "Cement and Concrete.")

Number	Age of Briquettes		Tensile Strength, Pounds per Square Inch				
		Per Cent Portland	100	50	25	12.5	0
		Per Cent Natural	0	50	75	87.5	100
1......	7 days	....	291	205	108	75	24
2......	28 days	....	357	264	219	190	123
3 . .	6 months	....	550	425	378	300	322
4......	1 year	....	574	441	360	336	291
5......	2 years	....	543	449	375	343	393
6	3 years	....	592	501	428	370	429

Mixture—1 cement : 2 "Point aux Pins" sand, passing No. 10 sieve.
Each result mean of 10 tests.

it was not until 1896 that a patent was granted to Mr. Jasper Whiting, of the Illinois Steel Company, for the production of this material in America. That year, this company made over 12,000 barrels of slag cement, while in 1903, the production amounted to 525,896 barrels, made at seven plants, two in Alabama, and one each in Illinois, Maryland, New Jersey, Ohio and Pennsylvania.

Composition.—The method employed in preparing the slag for this cement is to plunge it when still in a molten state into water, which not only makes it granular, but also prevents the breaking down of the complex compounds into the simple ones which form on cooling, thus retaining its structure and giving to it pozzuolanic properties. Slag allowed to cool slowly possesses scarcely any of these properties and is unfit for cement. Another great advantage of this granulation is the driving off of the excess of sulphur in the form of hydrogen sulphide gas.

The slags employed for this material must be basic, and low in magnesia and sulphur. Prof. Tetmajer states* that slags in which the ratio of lime to silica is unity are not suitable, but that above this proportion their value increases with this ratio. He also states that the best results are obtained from slags having a ratio of alumina to silica of between 0.45 and 0.50, the best ratio for the ingredients being—silica : alumina : lime = 30 : 16 : 46.

According to M. Prost† the slags used generally for the making of cements may be very nearly represented by the formula 2 SiO_2, Al_2O_3, 3 CaO, while slags of the formula 2 SiO_2, Al_2O_3, 4 CaO, may be used if quenched rapidly. He also states that a considerable proportion of sulphur may be present and not deleterious to the cement, although this is opposed to the consensus of opinion.

The specifications employed by the Illinois Steel Company for slags suitable for cement are:

"Silica plus alumina, not over 49 per cent.
Alumina—13 to 16 per cent.
Magnesia—not over 4 per cent.

"Slag must be made in a hot furnace, and must be of a light gray color.

"Slag must be thoroughly disintegrated by the action of a large stream of cold water directed against it with considerable force. This contact should be made as near the furnace as is possible."

The sulphur content is commonly restricted to 1.25 per cent. The amount of slaked lime to be added depends upon the composition of the slag and may vary from 5 to 30 per cent. of the finished product. It is customary in many plants to add a small proportion of caustic soda, from ¼ to 3 per cent., for the purpose of accelerating the set, the soda being usually dissolved in the water in which the lime is slaked, and thus added to the material. Analyses of several slags, and the cements made from them are shown in Table LVI.

*Annales de les Construction, July, 1886.
†Annales des Mines, 1889.

TABLE LVI.—The Composition of Slags Used for Pozzuolana Cements, and also of the Finished Product.

(From Eckels' "Cement Materials and Industry.")

LOCALITY	Material	Silica $Si\,O_2$	Alumina $Al_2\,O_3$	Iron Oxides $Fe\,O, Fe_2\,O_3$	Lime $Ca\,O$	Magnesia $Mg\,O$	Sulphuric Acid $S\,O_3$	Sulphur S	Sulphide of Lime Ca S	Loss on Ignition
Middlesborough—England	S'a ;	30.00	28 00	0 75	32.75	5.25	....	....	1 90	....
" "	"	31.50	18.56	....	42.22	3.18	2.21	0.45	....	....
Bilbao—Spain	"	32.90	13.25	0.46	47.30	1 37	....	....	3.42	....
" "	Cement	30.56	13 31	0.25	45.01	2.96	(a) 1.41	(b) 4 63	....	....
Saulncs—France	Slag	31.50	16 62	0.62	46 10	....	....	....	....	....
" "	Cement	22 45	13 95	3 30	51 10	1.35	0.35	....	....	7.50
Choindez—Switzerland	Slag	26.88	24.12	0.44	45 11	1.09	....	...	1.86	....
" "	"	27 33	23.81	0 63	45.83	0.92	....	....	1 34	....
" "	"	26 24	24 74	0 49	46.83	0.88	....	....	0.59	....
" "	Cement	19.50	17 50	....	54.00	....	....	....	....	....
Chicago—Illinois	Slag	32.20	15.50	....	48.14	2.27	....	....	....	....
" "	"	33.10	12 60	....	49 98	2.45	....	....	....	....
" "	"	31.80	14 80	....	49.74	2 29	....	....	....	....
" "	"	34.30	14.76	....	48.11	2 66	....	....	....	....
" "	Cement	27.20	14.18		50.03	3.22	0.15	....	....	4.25
" "	"	28 40	12.80		51.50	....	....	1.40	....	....
" "	"	28 95	11.40	0 54	50 29	2.96	....	1.37	....	3.39
" "	"	29.80	12.30		51.14	2.34	....	1 37	....	2.60
" "	"	27.80	11.10		50.96	2 23	....	1.18	....	5.30
N. Birmingham—Alabama	"	27.00	12.00		55 00	....	....	....	....	....
Ensley—[illegible]labama	"	27.78	11 70		51.71	1.39	....	1.31	....	....

(a) = $Ca\,So_4$ (b) = $Ca\,S$

Manufacture.—The following description of the manufacture of slag cement is taken from a statement* of the process made by Jasper Whiting, of the Illinois Steel Company, and is typical of the customary methods:

"Slag of the proper composition is made to flow from the furnace in which it is produced through an open trough to the chilling tank, where a large stream of cold water under high pressure is directed against it at right angles to its flow. Contact between the slag and cold water not only causes the slag to break up and disintegrate, but eliminates about one-third of its sulphur and changes it chemically in such a way as to render it suitable for the manufacture of cement.

"After each lot of slag is chilled a sample of it is taken and examined both chemically and physically in the laboratory. If the results of this examination are satisfactory, another sample is taken and mixed with a definite proportion of prepared lime and the whole ground in a miniature mill where actual cement is produced, which is also submitted to physical and chemical tests.

"The preliminary examination of the raw materials being complete, the slag is then passed through a dryer and conveyed by an elevator into bins located over grinding mills of the Griffin type, which are used for preliminary pulverization. It is then conveyed by means of another elevator into bins over grinding machines of the tube-mill type, where it is mixed with the proper proportions of prepared lime, as previously determined, and the two materials ground and intimately mixed together in the above mentioned tube-mills. The resulting powder, which is so fine that not over 4 per cent. is left on a 200-mesh sieve, is conveyed by means of screws and elevators into large bins, from which it is drawn and packed into barrels and bags for the market.

"An important element in the manufacture of cement is the prepared lime. This lime, obtained from the calcination of limestone, is unloaded into bins beneath which are placed two screens of different mesh, the coarse at the top. A quantity of lime is drawn on the top screen, where it is slaked by means of the addition of water containing in solution a small percentage

*Report of Board of Engineers U. S. A. on Steel Portland Cement, 1900, Appendix L.

of caustic soda. As the material is slaked it falls through the coarse screen on to the finer screen, after passing which it falls into a conveyor and is conveyed to a rotary dryer. It would be perfectly possible to slake this lime and incorporate with it the desired quantity of caustic soda without any additional heat, but by so doing there is great danger of having present particles of unslaked lime, which would render the resulting cement unfit for use. The wet and perfectly slaked lime ,therefore, is conveyed into the aforementioned dryer, and the final slaking done by the application of heat, so that every particle of lime is thoroughly slaked and the soda incorporated with it in the most perfect manner. The resulting prepared lime is then conveyed by means of elevators and screws into hoppers over the tube mills, where it is mixed with the ground slag in known but varying proportions."

Color and Weight.—Slag cement may usually be recognized by its bluish color and its very light weight, neither of which characteristics, however, may be considered as criteria of quality. The cement is packed in bags or barrels, which should weigh not less than 82½ and 330 pounds respectively, four of the bags equalling a barrel.

Specific Gravity.—Since the ingredients of this material are not burned together, the specific gravity of the cements equals the sum of the specific gravities of the ingredients (2.0 to 2.3 and 2.8 to 3.0), thus averaging from 2.6 to 2.85. Some specifications require a specific gravity of not less than 2.7, while others place both a maximum and minimum limit of, say, between 2.7 and 2.8, although the results of this test give but little indication of the constructive value of the material. The results tend to decrease with the age of the cement, due to the absorption of carbonic acid by the lime.

Fineness.—To produce a proper reaction on the addition of mixing water, it is necessary that slag cements be ground to a much greater degree of fineness than is necessary with Portlands, thus rendering them more susceptible to exterior conditions, some of which are beneficial while others are deleterious. Common practice is to require not less than 97% to pass the No. 100, and from 90 to 92% to pass the No. 200 sieve.

Time of Setting.—Most slag cements, made of a simple mixture of lime and slag, set so very slowly that they are difficult to use in practice, and hence it is customary to add a small percentage of caustic soda to the water in which the lime is slaked, thus increasing the rate of setting. If stored for a long time, however, the soda becomes carbonized and its effect disappears, the cement thus becoming slower setting with age. The specification requirements are generally similar to those for Portland cements, but it is especially important that the hard set requirement (usually 10 hours) be fulfilled with this material.

In spite of the fine grinding of slag cements, the amount of water required to bring them to normal consistency will average about 18% or 2 to 3% less than Portlands; otherwise the conduct of the test is similar.

Tensile Strength.—The strength of slag cements is tested in briquettes, both of neat paste and of sand mortar, made usually in the proportion of one part of cement to three parts of sand. Owing to the extreme fine grinding, the neat results are apt to be low, with the sand abnormally high, occasionally even equalling the neat. The tendency of the material prior to test is to decrease in strength with age, owing to the carbonization of the lime, while most other exterior conditions operate in a manner similar to Portland cements. In general, the results of the sand tests equal those of Portland cements, while the neat values are lower. The specifications* of the U. S. Army require 350 and 500 pounds neat, and 140 and 220 pounds with 1 : 3 mortar at the end of 7 and 28 days respectively. The report† accompanying these specifications states: "Mortars and concretes made from Puzzolan approximate in tensile strength similar mixtures of Portland cement, but their resistance to crushing is less, the ratio of crushing to tensile strength being about 6 or 7 to 1 for Puzzolan, and 9 to 11 to 1 for Portland."

Soundness.—The most important elements that may operate to produce unsoundness in slag cements are unslaked lime and excess of sulphides or magnesia. If the lime is not sufficiently slaked, or is coarsely ground, it will tend to produce swelling and disintegration, as with Portlands. The effect of sulphur in the form of sulphides is noticeable chiefly in air, where they oxidize to sulphates with a great change in volume, thus causing

*See Appendix D.
†Professional papers No. 28, Corps of Engineers, U. S. A.

disintegration; in water this change does not occur, although the pats generally show blotches of a bluish or greenish gray, due probably to the formation of iron sulphide.

The tests are made on normal pats and on specimens submitted to boiling, and should at the end of 28 days give, in the normal pats, no indication of unsoundness other than blotching, and should also pass the boiling test. Failure in either case should mean rejection.

Chemical Analysis.—Analysis of the essential ingredients gives little or no indication of the quality of slag cements. An excess of magnesia or of sulphur in the form of sulphides may produce unsoundness, and hence these ingredients should be limited to 4 and 1.3 per cent. respectively.

Testing of Slag Cements.—The methods of testing slag cements are practically identical with those for Portlands, the only noticeable difference being the smaller amount of water required to produce normal consistency. The methods of approximate testing given in Chapter XIII. are also entirely applicable to this material.

The essential tests are those of strength and soundness. Of lesser importance are fineness and time of setting, while that of specific gravity is of almost no value. It is advisable that the amount of sulphide sulphur always be determined if possible and never allowed to exceed 1.3 per cent. The specifications of the U. S. Army Engineers for this material are given in Appendix D.

Adaptability.—Slag cements are well suited for constructions in sea-water or in heavy foundations or other constructions such as sewers, conduits or other underground work, where constantly exposed to moisture. They, however, are not suited for any construction much exposed to the air, for under such conditions they are liable to disintegrate by reason of the oxidation of the sulphides, nor for work subject to either wearing or shocks, even when subjected to moisture. The white efflorescence usually appearing on the surface of slag cement concretes is an additional reason for not using this material where the appearance of the structure might thus be marred.

SAND CEMENT.

This material is produced by the fine grinding of an intimate mixture of sand and Portland cement, usually in equal proportions, although mixtures as lean as 1 cement to 6 sand have been made to compete with low grade natural cements. The sand should be clean and silicious, while the degree of fineness of the finished material must be such that at least 95 per cent. shall pass the No. 200 sieve.

Although neat briquettes of this cement are weaker than the Portland cement from which it is made, briquettes of 1 : 3 sand mortar give almost equal strength, due to the extreme fineness of the cement, which enables it to form a more perfect coating on the sand grains, and also to the fine particles of sand, which produce a better graded and hence denser mortar. The fine grinding of the cement also is beneficial in furthering the seasoning of any expansives that may be present.

The cement should be tested for strength (with sand only), soundness and time of setting, and should pass the specifications for Portland cement. The fineness should be such that 95% should pass the No. 200 sieve. A few tests made on sand cements of different proportions are shown in Table LVII. This cement is a patented article, but may be made by anyone on payment of a royalty. Mr. Sabin states* that "in the construction of Lock and Dam No. 2, Mississippi River, this process was used, grinding with a tube mill one part of Portland cement with one part fine sand. The cost, exclusive of plant, was estimated as follows:

½ barrel of Portland cement at $2.85......	$1.42
½ barrel of sand at $0.05	.03
Cost of grinding	.50
Cost of royalty	.05
Cost of one barrel sand-cement	$2.00"

thus effecting a saving of $0.85 cents per barrel less interest and depreciation of plant.

MIXED CEMENTS.

These are products resulting from the blending of Portland cements with raw rock, slag and natural cement, or from other

*In "Cement and Concrete," by L. C. Sabin.

TABLE LVII.—The Strength of Various Mixtures of Sand Cement. (Data from a Writer in "Engineering News," April 16, 1896.)

Mixture by Weight			Mixture Contains		CRUSHING STRENGTH—Pounds per Square Inch									
Sand Cement		*			In Water					In Air				
Cement	Sand	Sand	Cement	Sand	7 days	28 Days	3 mos.	6 mos.	1 year	7 days	28 Days	3 mos.	6 mos.	1 year
1	0	3	1	3	1595	2360	3420	.....	5055	1640	2500	3160	.....	3720
1	0	3	1	3	1165	1610	2150	.....	2967	1280	1870	2400	.....	3295
1	0	6	1	6	426	655	868	.....	1292	526	952	1110	.....	1760
1	2	2	1	8	1080	1795	2148	2578	3365	1122	2150	1565	3330	2911
1	3	2	1	11	497	1080	1637	2008	2485	610	1575	1408	2022	3536
1	6	2	1	20	327	555	1025	1117	1363	398	880	967	1209	1292
1	12	2	1	38	171	384	726	796	866	256	610	654	768	767
1	24	2	1	74	128	270	370	370	383	100	298	248	271	213
1	6	0	1	6	512	895	1380	.....	2357	600	1120	1460	.....	2258
1	6	1½	1	16½	370	540	852	.....	1576	440	940	960	.....	1533
1	6	2	1	20	200	384	667	.....	1050	256	682	725	.....	1079
1	6	2½	1	23½	142	284	540	.....	738	200	568	583	.....	710
1	6	3	1	27	114	228	426	.....	582	171	455	468	.....	610
1	3	2	1	11	497	1080	1637	2008	2485	612	1595	1408	2022	3496
1	3	2	1	11	640	1500	2136	2702	3081	625	1780	1908	3160	3081
1	3	2	1	11	670	1220	1651	2462	2670	725	1025	1671	2802	2868
					TENSILE STRENGTH—Pounds per Square Inch									
1	0	3	1	3	256	327	412	.....	500	270	370	455	.....	611
1	0	3	1	3	213	284	356	.....	356	284	370	426	.....	582
1	0	6	1	6	85	128	185	.....	200	114	185	213	.....	221
1	2	2	1	8	142	242	299	384	400	185	356	356	526	526
1	3	2	1	11	114	185	228	271	326	128	256	256	313	440
1	6	2	1	20	71	128	185	228	242	114	242	242	242	242
1	12	2	1	38	43	114	128	157	142	85	157	142	148	156
1	24	2	1	74	28	57	57	57	57	43	71	57	57	57
1	6	0	1	6	85	114	185	.....	312	114	185	185	.....	213
1	6	1½	1	16½	71	114	185	.....	298	100	200	200	.....	213
1	6	2	1	20	57	114	156	.....	220	85	171	171	.....	156
1	6	2½	1	23½	43	86	128	.....	128	85	128	114	.....	114
1	6	3	1	27	43	71	114	.....	142	71	85	85	.....	99
1	3	2	1	11	114	185	228	271	326	128	256	256	313	440
1	3	2	1	11	142	242	280	327	356	157	327	270	456	460
1	3	2	1	11	142	213	265	327	376	200	313	270	449	500

* Not Ground

artificial mixtures. They are commonly sold either as "second grade" Portlands or as natural cements. They exist in so many different varieties that it is impossible to consider or discuss them as a class. Tests for strength, soundness and time of setting should be made on them, the other tests affording but little information. Cements of this kind should never be employed in high grade construction, but occasionally may be used to advantage in rough foundation work, or in other cases where the best grades of material are not essential.

CHAPTER XVI.

SPECIFICATIONS.

The requirements for a good cement specification, or indeed for the specification of any material, are, first, that it be sufficiently severe to insure good material without containing requirements that unnecessarily hamper the manufacturer; second, that it be definite or free from ambiguous clauses, and, third, that it be well balanced.

The clauses of a cement specification should cover requirements for the storage and inspection of the shipments, the condition and weight of the packages, the method of testing to be followed, a definition of the material, the values to be obtained in the various tests, and the regulations regarding the acceptance or rejection of the shipments. The tests to which a cement should be subjected are specific gravity, fineness, time of setting, tensile strength neat and with sand, soundness, and the chemical determination of certain ingredients.

Although in the past there have been frequent examples of specifications of unnecessary severity, such cases are much less common than heretofore, thanks to the work of various technical societies in formulating standard specifications, and in bringing before the public, information regarding the values which should be obtained on a normal cement from each of these tests. Probably the most popular fallacy regarding the testing of cement is that great strength necessarily implies increased structural value, and hence there is a tendency on the part of certain engineers who have not taken the trouble to investigate the subject, to increase the requirements for tensile strength, with the purpose of securing an especially fine grade of material, when in fact the material furnished under such specifications is generally over-limed and plastered and, while giving high short time tests, will ultimately be inferior to the lower testing material, and also is much more susceptible to influences causing disintegration. Too low a requirement, on the other hand, will permit the use of an inferior or adulterated product, which in service may be insufficient to meet the demands required of the structure.

The same principle applies to all the other tests to which the cement may be subjected, an abnormally high specification requiring a forced product liable to give unsatisfactory service, and a low one permitting the use of inferior material, so that it is only by a most careful study of the available data that an original specification can be drafted that will ensure the furnishing of the best material.

While, as has been stated, the average qualities which a normal cement must possess are becoming much better understood, there is nevertheless in many recent specifications a lack of definiteness which is often sufficient to almost entirely destroy their value. First and foremost is the common omission of any clause or reference indicating the method to be employed in making the various tests. It has been shown in the previous chapters of this book what a great influence the methods and appliances used have upon the final results, and yet the majority of specifications require in, say, tensile strength, that the material shall develop a neat strength of, say, 500 pounds, at 7 days without any further qualifications, although it may easily be possible to obtain results of from 300 to 800 pounds according to the manner in which the material is treated. Although the method followed exerts probably a greater influence on tensile strength than on the other determinations, it nevertheless does apply to all of them, although in varying amounts, so that practically, unless a definite method of testing be stipulated, the acceptance of the material depends actually more upon the quality of the tester than on that of the cement itself.

For the average consumer, especially one just organizing a laboratory for the first time, it is best to stipulate in the specifications that the methods to be followed shall be in accordance with one of the standard methods, preferably that of the Committee of the American Society of Civil Engineers. In one particular, however, that of standard sand, the recommendations of this Committee are followed but rarely, so that it will generally be necessary to qualify the method in that detail if any other sand is to be used. The steaming test, moreover, has been found by all those who have investigated it in other than a superficial manner to be inferior to the boiling test in the reliability of its indications. Most consumers, therefore, should alter the method in these two particulars, but otherwise follow it implicitly in the conduct of the various tests. A clause to the following effect, therefore, will at once place the specification on a definite and standard basis, and will to a great extent preclude the rejection of good material, or the acceptance of bad material by reason of irregularities in the making of the determinations:

"All tests shall be made in accordance with the methods prescribed by the Committee on Uniform Tests of Cement of the American Society of Civil Engineers, reported January 21, 1903, and amended January 20, 1904, except in the following two particulars:

"(1) Sand of crushed quartz shall be substituted for Ottawa sand, the standard size remaining the same.

"(2) For the 'steam' test shall be substituted the boiling test, hereinafter described."

An even better method to be followed in drafting specifications for which the tests will be made in an established, well-equipped laboratory, is to stipulate that "all tests shall be made in accordance with the standard methods of testing now on file in the office of the Chief Engineer," these methods containing an exact description of every appliance and detail of the processes employed. The reason for this is that a well-organized laboratory will discover many small variations from the standard method that will much facilitate the routine of testing without in any way affecting the results to an appreciable extent. Now, if the standard method is specified and any of these variations is employed, the whole series of tests may be discredited and with reason, whereas if the laboratory has a record of its standard method which is specified and followed in practice, no such question can be raised. It is advised, however, that except for the use of the sand and the form of accelerated test recommended, that the methods specified conform as closely as possible to the report of the Committee, and thus arrive at as nearly standard methods as can be.

If no such method be stipulated, the specifications must contain complete information as to the essential points in the conduct of the tests, especially the normal consistency employed, the method of making and handling briquettes and pats, and descriptions of the apparatus, for unless this is done any specification, no matter how well drawn, is almost valueless.

Lack of definiteness in two other clauses also is often the cause of much annoyance—that relating to the facilities to be provided for inspection and testing, and that assigning the power of rejection to a certain individual. These provisions should be especially clear to prevent misunderstanding or friction.

The foregoing criticisms, it must be acknowledged, apply rather more to specifications of the past than the present since the wide publicity given in recent years to this subject has done much to promote greater familiarity with the essentials for good specifications. One fault, however, that still exists in many specifications is a lack of balance between the various requirements, which is apt to call for material almost impossible to produce, or to destroy the value of the other requirements. As a rule, specifications of this character show indications of having been drafted with a pair of shears, by an engineer who fully understood the purpose of the various requirements, but was unfamiliar with their inter-relations.

For example, a specification recently issued by the engineer of a city in the middle-west called for a slow-setting Portland cement, one finer than usual, and at the same time limited to content of sulphuric acid 1%, which would be a material practically impossible for most mills to produce. The engineer undoubtedly understood that a fine, slow setting cement was superior, and knew that high sulphuric acid might be deleterious, and having seen a specification, possibly of the French Government, limiting this element to 1%, he combined the three requirements, intending to secure an especially fine grade of material, but in reality precluding the use of some of the best cements. As it also happened the nature of the work was such that even an abnormal amount of sulphuric acid would not have affected its structural value.

Another "cribbed" clause often found is one limiting the content of magnesia in Portland cements to 2%, which may be met by certain brands, but which at one stroke practically eliminates all the excellent cements of the Lehigh Valley District, and at the same time does not secure better material. Another instance of lack of balance is frequently found in the fineness requirements, the author having had brought to his notice specifications for Portland cement, one calling for a residue of less than 15% on the No. 100 and 25% on the No. 200 sieve, and the other 8% on the No. 100 and 30% on the No. 200, the No. 100 requirement in the first case and the No. 200 in the second being almost worthless. The same lack of balance often occurs between the specifications for setting and fineness, fineness and strength, strength neat and strength with sand, etc., thus destroying much of their value. An engineer, unless well posted in the properties of cement, should therefore never attempt to combine the clauses from several specifications into one, but should either adopt one in its entirety or drop it altogether.

The recently published and most excellent standard specifications* issued by American, English and Canadian societies and also by such bodies as the Corps of Engineers of the U. S. Army have done much to better this condition. Considered only in the light of a clear, consistent and well-balanced specification, that of the Corps of Army Engineers ranks easily first, although the methods of testing employed are often unscientific in character and leave much to be desired. Those of the Committee of the American Society of Testing Materials are generally excellent, but are defective in the indefiniteness of the strength requirements, and also in the extremely low minimum values recommended, especially when it is considered that Ottawa sand and not crushed quartz is used.

Although it may be considered unwise to propose another specification, in view of the existence of these standards, it nevertheless cannot be denied that neither of these specifications is adapted to use exactly as it stands, the methods of testing in one instance, and the poor strength requirements in the other, leaving much to the discretion of the engineer, whose experience in these lines may not have been sufficient to properly make the necessary corrections. The following, therefore, is given as a cement specification which may be incorporated directly into the general specifications for construction work, and which, while insuring the furnishing of first-class material, will at the same time require only a normal product which any reputable manufacturer would be entirely willing to furnish, and moreover will not limit competition to any class of cements, nor to those produced by any particular process or in any special locality.

* See Appendices.

SPECIFICATIONS FOR PORTLAND CEMENT.*

1. **Definition.**—The cement shall be Portland cement of the best quality, dry and free from lumps. By Portland cement is meant the finely pulverized product resulting from the calcination to incipient fusion of an intimate mixture of properly proportioned argillaceous and calcareous materials, and to which no addition greater than 3% has been made subsequent to calcination.

2. **Inspection.**—All cements shall be inspected, and those rejected shall be immediately removed by the contractor. Every facility shall be provided by the contractor and a period of at least twelve days allowed for the inspection and necessary tests. Cement failing to meet the seven day requirements may be held awaiting the results of the twenty-eight day tests before rejection.

3. **Storage.**—While awaiting the results of the tests, the cement shall be stored in a suitable weather-tight building, having the floor properly blocked or raised from the ground, and shall be so stored as to permit easy access for the proper inspection and identification of each shipment.

4. **Packages.**—Cement shall be packed in strong cloth or canvas sacks, or in sound barrels lined with paper, which shall be plainly marked with the brand and the name of the manufacturer.†

5. **Weight.**—A barrel of cement shall contain 4 bags and shall weigh not less than 376 pounds net. A bag shall contain not less than 94 pounds net of cement. The weights of the separate packages shall be uniform.

6. **Tests.**‡—All tests shall be made in accordance with the methods prescribed by the Committee on Uniform Tests of Cement of the American Society of Civil Engineers, reported January 21, 1903, and amended January 20, 1904, except in the following two particulars:

(1) Sand of artificially prepared crushed quartz of the same size shall be substituted for Ottawa sand.

*These specifications are taken largely from the various standard specifications, as well as those of municipalities, and important engineering constructions.

†If the specifications are for the direct purchase of cement, there should be added to this clause:—"Packages received in broken or damaged condition may be rejected or accepted as fractional packages."

‡Or the following clause may be substituted for (6):—"All tests shall be made in accordance with the standard methods now on file in the office of the Chief Engineer, copies of which may be had on application."

(2) The boiling test, hereinafter described, shall be substituted for the "steam" test.

7. **Acceptance.**—The acceptance or rejection of a cement shall rest with the Chief Engineer, and shall be based upon the following requirements:

8. **Specific Gravity.**—The specific gravity of the cement shall be not less than 3.10.

9. **Fineness.**—It shall leave a residue of not more than 8% by weight on the No. 100, and not more than 25% on the No. 200 sieve.

10. **Time of Setting.**—It shall develop initial set in not less than 20 minutes, and must develop hard set within 10 hours.

11. **Tensile Strength.**—Briquettes one inch square in cross section shall develop not less than the following tensile strengths and shall show no retrogression in strength within the periods specified:

NEAT CEMENT.

Age.	Strength.
24 hours (in moist air)	175 lbs.
7 days (1 day in moist air, 6 days in water)	500 "
28 days (1 day in moist air, 27 days in water)	600 "

ONE PART CEMENT, THREE PARTS STANDARD SAND.

7 days (1 day in moist air, 6 days in water)	170 lbs.
28 days (1 day in moist air, 27 days in water)	240 "*

12. **Soundness.**—Two pats of neat cement of normal consistency about 3 ins. in diameter, one-half inch thick at the center and tapering to thin edges, and a ball of the same material about 1¼ ins. in diameter, shall be kept in moist air for a period of 24 hours.

(a) A pat is then kept in air at normal temperature, and observed at intervals for at least 28 days.

(b) A pat is kept in water maintained as near 70° Fahr. as practicable, and observed at intervals for at least 28 days.

*Additional security may be attained by specifying a maximum neat strength of 850 lbs. at seven days, and also an increase in the sand strength between the two periods of not less than 10%. Except for constructions of unusual importance, however, this clause is not recommended.

(c) The ball is placed in water at normal temperature, which is gradually (in about half an hour) raised to boiling and maintained there for 3 hours.

The pats, to pass the requirements satisfactorily, shall remain firm and hard and show no signs of distortion, blotching, checking, cracking or disintegration. The ball when removed from the water shall show no signs of checking, cracking, or disintegration.

13. **Chemical Requirements.**—The cement shall not contain more than 1.75% of anhydrous sulphuric acid (SO_3), nor more than 4% of magnesia (MgO).

SPECIFICATIONS FOR NATURAL CEMENT.

1. **Definition.**—The cement shall be natural cement of the best quality, dry and free from lumps. By natural cement is meant the finely pulverized product resulting from the calcination of an argillaceous limestone at a temperature below that necessary to cause incipient fusion.

2. **Inspection.**—All cements shall be inspected, and those rejected shall be removed immediately by the contractor. Every facility shall be provided by the contractor and a period of at least twelve days allowed for the inspection and necessary tests. Cement failing to meet the seven day requirements may be held awaiting the results of the twenty-eight day tests before rejection.

3. **Storage.**—While awaiting the results of the tests, the cement shall be stored in a suitable weather-tight building, having the floor properly blocked or raised from the ground, and shall be so stored as to permit easy access for the proper inspection and identification of each shipment.

4. **Packages.**—Cement shall be packed in strong cloth or canvas sacks, or in sound barrels lined with paper, which shall be plainly marked with the brand and the name of the manufacturer.*

5. **Weight.**—A barrel of cement shall contain 3 bags and shall weigh not less than 282 pounds net. A bag shall contain not less than 94 pounds net of cement. The weights of the separate packages shall be uniform.

*See foot note to clause (4)—Portland Cement, page 280.

6. **Tests.***—All tests shall be made in accordance with the methods prescribed by the Committee on Uniform Tests of Cement of the American Society of Civil Engineers, reported January 21, 1903, and amended January 20, 1904, except that sand of artificially prepared crushed quartz of the same size shall be substituted for Ottawa sand.

7. **Acceptance.**—The acceptance or rejection of a cement shall rest with the Chief Engineer, and shall be based upon the following requirements:

8. **Specific Gravity.**—The specific gravity of the cement shall not be less than 2.80.

9. **Fineness.**—It shall leave a residue of not more than 15% by weight on the No. 100, and not more than 30% on the No. 200 sieve.

10. **Time of Setting.**—It shall develop initial set in not less than 10 minutes, and hard set in not more than 5 hours.

11. **Tensile Strength.**—Briquettes one inch square in cross section shall develop not less than the following tensile strengths and shall show no retrogression in strength within the periods specified.

NEAT CEMENT.

Age.	Strength.
24 hours (in moist air)	40 lbs.
7 days (1 day in moist air, 6 days in water)	125 "
28 days (1 day in moist air, 27 days in water)	225 "

ONE PART CEMENT, TWO PARTS STANDARD SAND.

7 days (1 day in moist air, 6 days in water)	75 lbs.
28 days (1 day in moist air, 27 days in water)	140 "

12. **Soundness.**—Two pats of neat cement of normal consistency about 3 ins. in diameter, ½-in. thick at the center and tapering to thin edges shall be kept in moist air for a period of 24 hours.

(a) A pat is then kept in air at normal temperature.

(b) The other pat is kept in water maintained as near 70° Fahr. as practicable.

These pats shall be observed at intervals for at least 28 days, and to satisfactorily pass the requirements shall remain firm and

*See foot note to clause (6)—Portland Cement, page 280.

hard, and show no signs of distortion, checking, cracking or disintegration.

INTERPRETATION OF SPECIFICATIONS.

In the interpretation of the results of specification tests, it must always be borne in mind that the cement should be judged from the results of all the tests collectively, and not from the individual values. It can be stated that only failure in the normal pat tests, or abnormally low values in the sand strength, is sufficient to warrant the rejection of the shipment, without other evidences of poor quality. For example, let us consider the ten Portland cements in Table LVIII., all of which occurred in the routine of the author's laboratory, it being assumed that the specifications just given formed the basis of the testing.

Cement 1 is normal in every particular and one which would be entirely safe to accept on the seven day test. The second cement fails in specific gravity, and is slightly below the specifications in sand strength. The boiling test, however, was good, and an examination showed neither underburning nor adulteration, thus indicating that the cement was merely old, as also was more or less apparent from its condition. The shipment, therefore, was accepted at 7 days, and the 28 day test confirmed this decision.

Cements 3 and 8 both gave evidences of being over-limed in failure in the boiling test and in the high neat strength at 7 days. Both of these shipments were held at 7 days, second boiling tests made at 28 days, the first passing and the second failing, and as a result the first sample was accepted while the second was rejected. At the end of 3 months the normal pats of No. 8 had completely disintegrated, while the others remained normal except for a very slight curvature. Cement 4 is another example of low sand strength and failure in boiling, which was accepted on the 28 day test.

Cements 5 and 6 are both coarse and fail to boil. At 28 days No. 5 boiled and No. 6 failed a second time. No. 5 was accepted, therefore, while No. 6 was rejected, but at the same time the manufacturer who furnished cement No. 5 was notified that the shipment was coarse, and that future shipments would be condemned if this were not rectified.

Cement 7 is extremely quick setting, and for most classes of construction should be rejected outright. No. 9 illustrates a

TABLE LVIII.—Typical Routine Tests of Portland Cement. (From Tests by the Author.)

	1	2	3	4	5	6	7	8	9	10
Specific Gravity...	3 143	3.084	3.152	3.182	3 149	3.143	3 125	3.140	3.092	3.074
Fineness No. 50...	0 0	0.0	0.0	0.0	0.0	0.5	0.0	0.0	0.0	0 6
" " 100...	7.6	7.0	7.4	8.0	10.2	12.6	6.4	7.2	11.3	5.8
" " 200...	23 5	22.8	24.0	24.6	27.6	31.5	22.4	24.3	27.6	20.4
Initial set.........	120	165	170	223	159	206	3	35	44	42
Hard set..........	315	374	355	435	364	391	25	212	193	231
TENSILE STRENGTH:										
Neat, 24 hours...	352	212	612	252	423	406	324	421	357	256
" 7 days....	710	580	854	612	712	702	719	892	618	625
" 28 "	805	692	793	735	819	784	594	654	722	753
1:3, 7 "	228	167	245	154	182	158	174	218	162	182
1:3, 28 "	301	261	312	243	249	219	232	226	221	257
NORMAL PATS.										
Air, 7 days......	O. K.	O. K.	Left glass.	O. K.	O. K.	O. K.	Left glass.	Expansion cracks.	Expansion cracks.	Blotched, left glass.
" 28 "	Left glass.	O. K.	Very slightly inside curled. Left glass.	O. K,	O. K.	Left glass.	Slightly inside curled. Left glass.	Inside curled. Left glass.	Expansion cracks.	Blotched, slightly, inside curled. Left glass.
Water, 7 days...	O. K.	O. K.	O. K.	Left glass.	O. K.	Left glass.	Left glass.	Left glass.	Left glass.	Blotched.
" 28 " ...	O. K.	O. K.	Very slightly curled. Left glass.	Left glass.	O. K.	Left glass.	Very slightly curled. Left glass.	Radial cracks. Left glass.	Very slightly curled. Left glass.	Blotched, very slightly curled. Left glass.
Boiling test.......	O. K.	O. K.	Disintegrated.	Checked and cracked.	Slightly checked.	Disintegrated.	O. K.	Disintegrated.	Checked.	O. K.

thoroughly bad cement, both underburned and coarse, while 10 shows a typical case of adulteration with slag, indicated by the low specific gravity, the blotching of the normal pats, as well as by the special tests for adulteration.

The point, however, which it is especially desired to emphasize is that while cements 2, 3, 4 and 5 all fail in some particular to pass the requirements of the specifications, all of them were accepted and proved satisfactory in service. To attempt to hold every shipment to pass an absolutely perfect test is not only unwarranted by experience, but often defeats its own purpose in rejecting excellent material, while it also creates much unnecessary annoyance and delay.

APPENDIX A.

Standard Method of Cement Testing.

Progress Report of the Committee of the American Society of Civil Engineers on Uniform Tests of Cement, presented January 21, 1903, and amended January 20, 1904.

Sampling.

1.—SELECTION OF SAMPLE.—The selection of the sample for testing is a detail that must be left to the discretion of the engineer; the number and the quantity to be taken from each package will depend largely on the importance of the work, the number of tests to be made and the facilities for making them.

2.—The sample shall be a fair average of the contents of the package; it is recommended that, where conditions permit, one barrel in every ten be sampled.

3.—All samples should be passed through a sieve having twenty meshes per linear inch in order to break up lumps and remove foreign material; this is also a very effective method for mixing them together in order to obtain an average. For determining the characteristics of a shipment of cement, the individual samples may be mixed and the average tested; where time will permit, however, it is recommended that they be tested separately.

4.—METHOD OF SAMPLING.—Cement in barrels should be sampled through a hole made in the center of one of the staves, midway between the heads, or in the head, by means of an auger or a sampling iron similar to that used by sugar inspectors. If in bags, it should be taken from surface to center.

Chemical Analysis.

5.—SIGNIFICANCE.—Chemical analysis may render valuable service in the detection of adulteration of cement with considerable amounts of inert material, such as slag or ground limestone. It is, of use, also, in determining whether certain constituents, believed to be harmful when in excess of a certain percentage, as magnesia and sulphuric anhydride, are present in inadmissible proportions. While not recommending a definite limit for these impurities, the Committee would suggest that the most recent and reliable evidence appears to indicate that, for Portland cement, magnesia to the amount of 5 per cent. and sulphuric anhydride to the amount of 1.75 per cent. may safely be considered harmless.

6.—The determination of the principal constituents of cement—silica, alumina, iron oxide and lime—is not conclusive as an indication of quality. Faulty character of cement results more frequently from imperfect preparation of the raw material or defective burning than from incorrect proportions of the constituents. Cement made from very finely-ground material, and thoroughly burned, may contain much more lime than the amount usually present and still be perfectly sound. On the other hand, cements low in lime may, on account of careless preparation of the raw material, be of dangerous character. Further, the ash of the fuel used in burning may so greatly modify the composition of the product as largely to destroy the significance of the results of analysis.

7.—METHOD.—As a method to be followed for the analysis of cement, that proposed by the Committee on Uniformity in the Analysis of Materials for the Portland Cement Industry, of the New York Section of the Society for Chemical Industry, and published in the Journal of the Society for January 15, 1902, is recommended.

Specific Gravity.

8.—SIGNIFICANCE.—The specific gravity of cement is lowered by underburning, adulteration and hydration, but the adulteration must be in considerable quantity to affect the results appreciably.

9.—Inasmuch as the differences in specific gravity are usually very small, great care must be exercised in making the determination.

10.—When properly made, this test affords a quick check for underburning or adulteration.

11.—APPARATUS AND METHOD.—The determination of specific gravity is most conveniently made with Le Chatelier's apparatus. This consists of a flask (D), Fig. 1 (see Fig. 138), of 120 cu. cm. (7.32 cu. ins.) capacity, the neck of which is about 20 cm. (7.87 ins.) long; in the middle of this neck is a bulb (C), above and below which are two marks (F) and (E); the volume between these marks is 20 cu. cm. (1.22 cu. ins.). The neck has diameter of about 9 mm. (0.35 ins.), and is graduated into tenths of cubic centimeters above the mark (F).

12.—Benzine (62 degrees Baumé naphtha), or kerosene free from water, should be used in making the determination.

13.—The specific gravity can be determined in two ways:

(1) The flask is filled with either of these liquids to the lower mark (E), and 64 gr. (2.25 oz.) of powder, previously dried at 100° Cent. (212° Fahr.) and cooled to the temperature of the liquid, is gradually introduced through the funnel (B) [the stem of which extends into the flask to the top of the bulb (C)], until the upper mark (F) is reached. The difference in weight between the cement remaining and the original quantity (64 gr.) is the weight which has displaced 20 cu. cm.

14.—(2) The whole quantity of the powder is introduced, and the level of the liquid rises to some division of the graduated neck. This reading plus 20 cu. cm. is the volume displaced by 64 gr. of the powder.

15.—The specific gravity is then obtained from the formula:

$$\text{Specific gravity} = \frac{\text{Weight of cement}}{\text{Displaced volume}}$$

16.—The flask, during the operation, is kept immersed in water in a jar (A), in order to avoid variations in the temperature of the liquid. The results should agree within 0.01.

17.—A convenient method for cleaning the apparatus is as follows: The flask is inverted over a large vessel, preferably a glass jar, and shaken vertically until the liquid starts to flow freely; it is then held still in a vertical position until empty; the remaining traces of cement can be re-

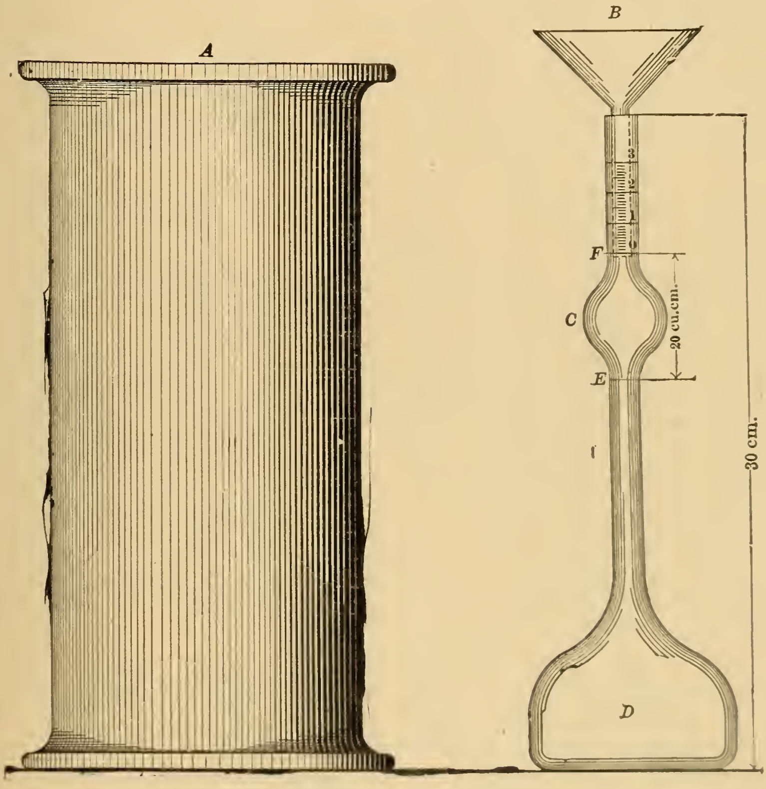


FIG. 138.—Le Chatelier's Specific Gravity Apparatus.

moved in a similar manner by pouring into the flask a small quantity of clean liquid and repeating the operation.

18.—More accurate determinations may be made with the picnometer.

Fineness.

19.—SIGNIFICANCE.—It is generally accepted that the coarser particles in cement are practically inert, and it is only the extremely fine powder that possesses adhesive or cementing qualities. The more finely cement is pulverized, all other conditions being the same, the more sand it will carry and produce a mortar of a given strength.

20.—The degree of final pulverization which the cement receives at the place of manufacture is ascertained by measuring the residue retained on certain sieves. Those known as the No. 100 and No. 200 sieves are recommended for this purpose.

21.—Apparatus.—The sieves should be circular, about 20 cm. (7.87 ins.) in diameter, 6 cm. (2.36 ins.) high, and provided with a pan 5 cm. (1.97 ins.) deep, and a cover.

22.—The wire cloth should be woven from brass wire having the following diameters:

No. 100, 0.0045 in.; No. 200, 0.0024 in.

23.—This cloth should be mounted on the frames without distortion; the mesh should be regular in spacing and be within the following limits:

No. 100, 96 to 100 meshes to the linear inch.
No. 200, 188 to 200 meshes to the linear inch.

24.—Fifty grams (1.76 oz.) or 100 gr. (3.52 oz.) should be used for the test, and dried at a temperature of 100° Cent. (212° Fahr.) prior to sieving.

25.—Method.—The Committee, after careful investigation, has reached the conclusion that mechanical sieving is not as practicable or efficient as hand work, and, therefore, recommends the following method:

26.—The thoroughly dried and coarsely screened sample is weighed and placed on the No. 200 sieve, which, with pan and cover attached, is held in one hand in a slightly inclined position, and moved forward and backward, at the same time striking the side gently with the palm of the other hand, at the rate of about 200 strokes per minute. The operation is continued until not more than one-tenth of 1 per cent. passes through after one minute of continuous sieving. The residue is weighed, then placed on the No. 100 sieve and the operation repeated. The work may be expedited by placing in the sieve a small quantity of large shot. The results should be reported to the nearest tenth of 1 per cent.

Normal Consistency.

27.—Significance.—The use of a proper percentage of water in making the pastes* from which pats, tests of setting and briquettes are made, is exceedingly important, and affects vitally the results obtained.

28.—The determination consists in measuring the amount of water required to reduce the cement to a given state of plasticity, or to what is usually designated the normal consistency.

29.—Various methods have been proposed for making this determination, none of which has been found entirely satisfactory. The Committee recommends the following:

30.—Method. Vicat Needle Apparatus.—This consists of a frame (K), Fig. 2 (see Fig. 139), bearing a movable rod (L), with a cap (A) at one end, and at the other the cylinder (B), 1 cm. (0.39 in.) in diameter, the cap, rod and cylinder weighing 300 gr. (10.58 oz.). The rod, which can be held in any desired position by a screw (F), carries an indicator, which moves over a scale (graduated to centimeters) attached to the frame (K). The paste is held by a conical, hard-rubber ring (I),

*The term "paste" is used in this report to designate a mixture of cement and water, and the word "mortar" a mixture of cement, sand and water.

7 cm. (2.76 ins.) in diameter at the base, 4 cm. (1.57 ins.) high, resting on a glass plate (J), about 10 c. m. (3.94 ins.) square.

31.—In making the determination, the same quantity of cement as will be subsequently used for each batch in making the briquettes (but not less than 500 grams) is kneaded into a paste, as described in Paragraph 58, and quickly formed into a ball with the hands, completing the operation by tossing it six times from one hand to the other, maintained 6 ins. apart; the ball is then pressed into the rubber ring, through the larger opening, smoothed off, and placed (on its large end) on a glass plate and the smaller end smoothed off with a trowel; the paste confined in the ring,

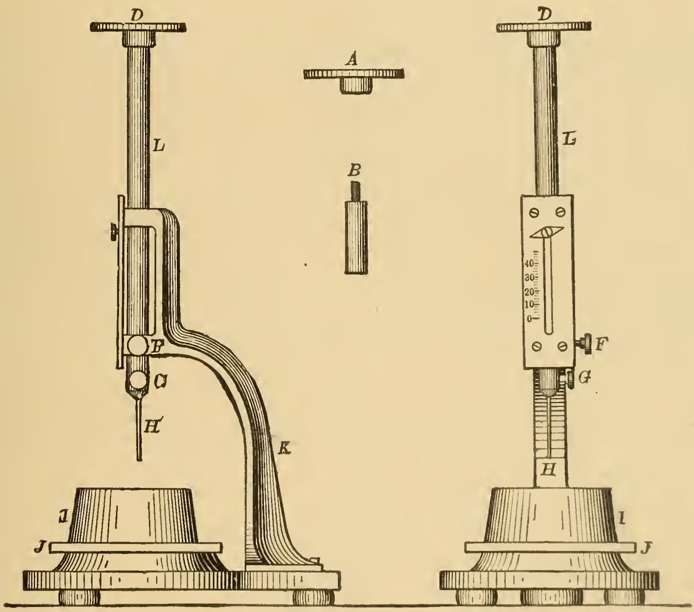


FIG. 139.—The Vicat Needle.

resting on the plate, is placed under the rod bearing the cylinder, which is brought in contact with the surface and quickly released.

32.—The paste is of normal consistency when the cylinder penetrates to a point in the mass 10 mm. (0.39 in.) below the top of the ring. Great care must be taken to fill the ring exactly to the top.

33.—The trial pastes are made with varying percentages of water until the correct consistency is obtained.

34.—The Committee has recommended, as normal, a paste, the consistency of which is rather wet, because it believes that variations in the amount of compression to which the briquette is subjected in moulding are likely to be less with such a paste.

35.—Having determined in this manner the proper percentage of water required to produce a paste of normal consistency, the proper percentage required for the mortars is obtained from an empirical formula.

36.—The Committee hopes to devise such a formula. The subject proves to be a very difficult one, and, although the Committee has given it much study, it is not yet prepared to make a definite recommendation.

Time of Setting.

37.—SIGNIFICANCE.—The object of this test is to determine the time which elapsed from the moment water is added until the paste ceases to be fluid and plastic (called the "initial set"), and also the time required for it to acquire a certain degree of hardness (called the "final" or "hard set"). The former of these is the more important, since, with the commencement of setting, the process of crystallization or hardening is said to begin. As a disturbance of this process may produce a loss of strength, it is desirable to complete the operation of mixing and moulding or incorporating the mortar into the work before the cement begins to set.

38.—It is usual to measure arbitrarily the beginning and end of the setting by the penetration of weighted wires of given diameters.

39.—METHOD.—For this purpose the Vicat Needle, which has already been described in Paragraph 30, should be used.

40.—In making the test, a paste of normal consistency is moulded and placed under the rod (L); Fig. 2, as described in Paragraph 31; this rod, bearing the cap (D) at one end and the needle (H), 1 mm. (0.039 in.) in diameter, at the other, weighing 300 gr. (10.58 oz.). The needle is then carefully brought in contact with the surface of the paste and quickly released.

41.—The setting is said to have commenced when the needle ceases to pass a point 5 mm. (0.20 in.) above the upper surface of the glass plate, and is said to have terminated the moment the needle does not sink visibly into the mass.

42.—The test pieces should be stored in moist air during the test; this is accomplished by placing them on a rack over water contained in a pan and covered with a damp cloth, the cloth to be kept away from them by means of a wire screen; or they may be stored in a moist box or closet.

43.—Care should be taken to keep the needle clean, as the collection of cement on the sides of the needle retards the penetration, while cement on the point reduces the area and tends to increase the penetration.

44.—The determination of the time of setting is only approximate, being materially affected by the temperature of the mixing water, the temperature and humidity of the air during the test, the percentage of water used, and the amount of moulding the paste receives.

Standard Sand.

45.—The Committee recognizes the grave objections to the standard quartz now generally used, especially on account of its high percentage of voids, the difficulty of compacting in the moulds, and its lack of uniformity; it has spent much time in investigating the various natural sands which appeared to be available and suitable for use.

46.—For the present, the Committee recommends the natural sand from Ottawa, Ill., screened to pass a sieve having 20 meshes per linear inch and retained on a sieve having 30 meshes per linear inch; the wires to have diameters of 0.0165 and 0.0112 in., respectively, i. e., half the width of the opening in each case. Sand having passed the No. 20 sieve shall be considered standard when not more than one per cent. passes a No. 30 sieve after one minute continuous sifting of a 500-gram sample.

47.—The Sandusky Portland Cement Company, of Sandusky, Ohio, has agreed to undertake the preparation of this sand, and to furnish it at a price only sufficient to cover the actual cost of preparation.

Form of Briquette.

48.—While the form of the briquette recommended by a former Committee of the Society is not wholly satisfactory, this Committee is not prepared to suggest any change, other than rounding off the corners by curves of ½-inch radius, Fig. 3 (see Fig. 43).

Moulds.

49.—The moulds should be made of brass, bronze or some equally non-corrodible material, having sufficient metal in the sides to prevent spreading during moulding.

50.—Gang moulds, which permit moulding a number of briquettes at one time, are preferred by many to single moulds; since the greater quantity of mortar that can be mixed tends to produce greater uniformity in the results. The type shown in Fig. 4 (see Fig. 46) is recommended.

51.—The moulds should be wiped with an oily cloth before using.

Mixing.

52.—All proportions should be stated by weight; the quantity of water to be used should be stated as a percentage of the dry material.

53.—The metric system is recommended because of the convenient relation of the gram and the cubic centimeter.

54.—The temperature of the room and the mixing water should be as near 21 degrees Cent. (70 degrees Fahr.) as it is practicable to maintain it.

55.—The sand and cement should be thoroughly mixed dry. The mixing should be done on some non-absorbing surface, preferably plate glass. If the mixing must be done on an absorbing surface it should be thoroughly dampened prior to use.

56.—The quantity of material to be mixed at one time depends on the number of test pieces to be made; about 1,000 gr. (35.28 oz.) makes a convenient quantity to mix, especially by hand methods.

57.—The Committee, after investigation of the various mechanical mixing machines, has decided not to recommend any machine that has thus far been devised, for the following reasons:

(1) The tendency of most cement is to "ball up" in the machine, thereby preventing the working of it into a homogeneous paste; (2) there are no means of ascertaining when the mixing is complete without stopping the machine, and (3) the difficulty of keeping the machine clean.

58.—METHOD.—The material is weighed and placed on the mixing table, and a crater formed in the center, into which the proper percentage of clean water is poured; the material on the outer edge is turned into the crater by the aid of a trowel. As soon as the water has been absorbed, which should not require more than one minute, the operation is completed by vigorously kneading with the hands for an additional 1½ minutes, the process being similar to that used in kneading dough. A sand-glass affords a convenient guide for the time of kneading. During the operation of mixing the hands should be protected by gloves, preferably of rubber.

Moulding.

59.—Having worked the paste or mortar to the proper consistency, it is at once placed in the moulds by hand.

60.—The Committee has been unable to secure satisfactory results with the present moulding machines; the operation of machine moulding is very slow, and the present types permit of moulding but one briquette at a time, and are not practicable with the pastes or mortars herein recommended.

61.—METHOD.—The moulds should be filled at once, the material pressed in firmly with the fingers and smoothed off with a trowel without ramming; the material should be heaped up on the upper surface of the mould, and, in smoothing off, the trowel should be drawn over the mould in such a manner as to exert a moderate pressure on the excess material. The mould should be turned over and the operation repeated.

62.—A check upon the uniformity of the mixing and moulding is afforded by weighing the briquettes just prior to immersion, or upon removal from the moist closet. Briquettes which vary in weight more than 3 per cent. from the average should not be tested.

Storage of the Test Pieces.

63.—During the first 24 hours after moulding the test pieces should be kept in moist air to prevent them from drying out.

64.—A moist closet or chamber is so easily devised that the use of the damp cloth should be abandoned if possible. Covering the test pieces with a damp cloth is objectionable, as commonly used, because the cloth may dry out unequally, and, in consequence, the test pieces are not all maintained under the same condition. Where a moist closet is not available, a cloth may be used and kept uniformly wet by immersing the ends in water. It should be kept from direct contact with the test pieces by means of a wire screen or some similar arrangement.

65.—A moist closet consists of a soapstone or slate box or a metal-lined wooden box—the metal lining being covered with felt and this felt kept wet. The bottom of the box is so constructed as to hold water, and the sides are provided with cleats for holding glass shelves on which to place the briquettes. Care should be taken to keep the air in the closet uniformly moist.

66.—After 24 hours in moist air the test pieces for longer periods of time should be immersed in water maintained as near 21° Cent. (70° Fahr.) as practicable; they may be stored in tanks or pans, which should be of non-corrodible material.

Tensile Strength.

67.—The tests may be made on any standard machine. A solid metal clip, as shown in Fig. 5 (see Fig. 76), is recommended. This clip is to be used without cushioning at the points of contact with the test specimen. The bearing at each point of contact should be ¼-in. wide, and the distance between the center of contact on the same clip should be 1¼ ins.

68.—Test pieces should be broken as soon as they are removed from the water. Care should be observed in centering the briquettes in the testing machine, as cross-strains, produced by improper centering, tend to lower the breaking strength. The load should not be applied too suddenly, as it may produce vibration, the shock from which often breaks the briquette before the ultimate strength is reached. Care must be taken that the clips and the sides of the briquette be clean and free from grains of sand or dirt, which would prevent a good bearing. The load should be applied at the rate of 600 lbs. per minute. The average of the briquettes of each sample tested should be taken as the test, excluding any results which are manifestly faulty.

Constancy of Volume.

69.—Significance.—The object is to develop those qualities which tend to destroy the strength and durability of a cement. As it is highly essential to determine such qualities at once, tests of this character are for the most part made in a very short time, and are known, therefore, as accelerated tests. Failure is revealed by cracking, checking, swelling or disintegration, or all of these phenomena. A cement which remains perfectly sound is said to be of constant volume.

70.—Methods.—Tests for constancy of volume are divided into two classes: (1) Normal tests, or those made in either air or water maintained at about 21° Cent. (70° Fahr.), and (2) accelerated tests, or those made in air, steam or water at a temperature of 45° Cent. (115° Fahr.) and upward. The test pieces should be allowed to remain 24 hours in moist air before immersion in water or steam, or preservation in air.

71.—For these tests, pats, about 7½ cm. (2.95 ins.) in diameter, 1¼ cm. (0.49 in.) thick at the center, and tapering to a thin edge, should be made upon a clean glass plate [about 10 cm. (3.94 ins.) square], from cement paste of normal consistency.

72.—Normal Test.—A pat is immersed in water maintained as near 21° Cent. (70° Fahr.) as possible for 28 days, and observed at intervals. A similar pat is maintained in air at ordinary temperature and observed at intervals.

73.—Accelerated Test.—A pat is exposed in any convenient way in an atmosphere of steam, above boiling water, in a loosely closed vessel, for 3 hours.

74.—To pass these tests satisfactorily, the pats should remain firm and hard, and show no signs of cracking, distortion or disintegration.

75.—Should the pat leave the plate, distortion may be detected best with a straight-edge applied to the surface which was in contact with the plate.

76.—In the present state of our knowledge it cannot be said that cement should necessarily be condemned simply for failure to pass the accelerated tests; nor can a cement be considered entirely satisfactory, simply because it has passed these tests.

Submitted on behalf of the Committee.

GEORGE S. WEBSTER,
Chairman.
RICHARD L. HUMPHREY,
Secretary.

Committee.
George S. Webster,

Richard L. Humphrey,	S. B. Newberry,
George F. Swain,	Clifford Richardson,
Alfred Noble,	W. B. W. Howe,
Louis C. Sabin,	F. H. Lewis.

APPENDIX B.

Standard Method for the Chemical Analysis of Cement.

Adopted by the New York Section of the Society for Chemical Industry, January, 1902.

Solution.

One-half gram of the finely-powdered substance is to be weighed out and, if a limestone or unburned mixture, strongly ignited in a covered platinum crucible over a strong blast for 15 minutes, or longer if the blast is not powerful enough to effect complete conversion to a cement in this time. It is then transferred to an evaporating dish, preferably of platinum for the sake of celerity in evaporation, moistened with enough water to prevent lumping, and 5 to 10 c. c. of strong HCl added and digested with the aid of gentle heat and agitation until solution is complete. Solution may be aided by light pressure with the flattened end of a glass rod.* The solution is then evaporated to dryness, as far as this may be possible on the bath.

Silica (SiO_2).

The residue without further heating is treated at first with 5 to 10 c. c. of strong HCl, which is then diluted to half strength or less, or upon the

*If anything remains undecomposed it should be separated, fused with a little Na_2CO_3, dissolved and added to the original solution. Of course a small amount of separated non-gelatinous silica is not to be mistaken for undecomposed matter.

residue may be poured at once a larger volume of acid of half strength. The dish is then covered and digestion allowed to go on for 10 minutes on the bath, after which the solution is filtered and the separated silica washed thoroughly with water. The filtrate is again evaporated to dryness, the residue without further heating, taken up with acid and water and the small amount of silica it contains separated on another filter paper. The papers containing the residue are transferred wet to a weighed platinum crucible, dried, ignited, first over a Bunsen burner until the carbon of the filter is completely consumed, and finally over the blast for 15 minutes and checked by a further blasting for 10 minutes or to constant weight. The silica, if great accuracy is desired, is treated in the crucible with about 10 c. c. of HFl and four drops of H_2SO_4 and evaporated over a low flame to complete dryness. The small residue is finally blasted, for a minute or two, cooled and weighed. The difference between this weight and the weight previously obtained gives the amount of silica.*

Alumina and Iron (AL_2O_3 and Fe_2O_3).

The filtrate, about 250 c. c., from the second evaporation for SiO_2, is made alkaline with NH_4OH after adding H Cl, if need be, to insure a total of 10 to 15 c. c strong acid, and boiled to expel excess of NH_3, or until there is but a faint odor of it, and the precipitate iron and aluminum hydrates, after settling, are washed once by decantation and slightly on the filter. Setting aside the filtrate, the precipitate is dissolved in hot dilute HCl, the solution passing into the beaker in which the precipitation was made. The aluminum and iron are then reprecipitated by NH_4OH, boiled and the second precipitate collected and washed on the same filter used in the first instance. The filter paper, with the precipitate, is then placed in a weighed platinum crucible, the paper burned off and the precipitate ignited and finally blasted 5 minutes, with care to prevent reduction, cooled and weighed as $Al_2O_3 + Fe_2O_3$.†

Iron (Fe_2O_3).

The combined iron and aluminum oxides are fused in a platinum crucible at a very low temperature with about 3 to 4 grams $KHSO_4$, or better $NaHSO_4$, the melt taken up with so much dilute H_2SO_4, that there shall be no less than 5 grams absolute acid and enough water to effect solution on heating. The solution is then evaporated and eventually heated till acid fumes come off copiously. After cooling and redissolving in water the small amount of silica is filtered out, weighed and corrected by HFl and H_2SO_4.‡ The filtrate is reduced by zinc, or preferably by hydrogen sulphide, boiling out the excess of the latter afterward while passing CO_2 through the flask, and titrated with permanganate.§ The strength of the permanganate solution should not be greater than .0040 gr. Fe_2O_3 per c. c.

---

*For ordinary control in the plant laboratory this correction may, perhaps, be neglected; the double evaporation never.

†This precipitate contains TiO_2, P_2O_5, Mn_3O_4.

‡This correction of Al_2O_3 Fe_2O_3 for silica should not be made when the HFl correction of the main silica has been omitted, unless that silica was obtained by only one evaporation and filtration. After two evaporations and filtrations 1 to 2 mg. of SiO_2 are still to be found with the Al_2O_3 Fe_2O_3.

§In this way only is the influence of titanium to be avoided and a correct result obtained for iron.

Lime (CaO).

To the combined filtrate from the $Al_2O_3 + Fe_2O_3$ precipitate a few drops of NH_4OH are added, and the solution brought to boiling. To the boiling solution 20 c. c. of a saturated solution of ammonium oxalate are added, and the boiling continued until the precipitated CaC_2O_4 assumes a well-defined granular form. It is then allowed to stand for 20 minutes, or until the precipitate has settled, and then filtered and washed. The precipitate and filter are placed wet in a platinum crucible, and the paper burned off over a small flame of a Bunsen burner. It is then ignited, redissolved in HCl, and the solution made up to 100 c. c. with water. Ammonia is added in slight excess, and the liquid is boiled. If a small amount of Al_2O_3 separates this is filtered out, weighed, and the amount added to that found in the first determination, when greater accuracy is desired. The lime is then reprecipitated by ammonium oxalate, allowed to stand until settled, filtered, and washed,* weighed as oxide by ignition and blasting in a covered crucible to constant weight, or determined with dilute standard permanganate.†

Magnesia (MgO).

The combined filtrates from the calcium precipitates are acidified with HCl and concentrated on the steam bath to about 150 c. c., 10 c. c. of saturated solution of $Na(NH_4)HPO_4$ are added and the solution boiled for several minutes. It is then removed from the flame and cooled by placing the beaker in ice water. After cooling. NH_4OH is added drop by drop with constant stirring until the crystalline ammonium-magnesium ortho-phosphate begins to form, and then in moderate excess, the stirring being continued for several minutes. It is then set aside for several hours in a cool atmosphere and filtered. The precipitate is redissolved in hot dilute HCl, the solution made up to about 100 c. c., 1 c. c. of a saturated solution of $Na(NH_4)HPO_4$ added ,and ammonia drop by drop, with constant stirring until the precipitate is again formed as described and the ammonia is in moderate excess. It is then allowed to stand for about 2 hours, when it is filtered on a paper or a Gooch crucible, ignited, cooled and weighed as $Mg_2P_2O_7$.

Alkalies (K_2O and Na_2O).

For the determination of the alkalies, the well known method of Prof. J. Lawrence Smith is to be followed, either with or without the addition of $CaCO_3$ with NH_4Cl.

Anhydrous Sulphuric Acid (SO_3).

One gram of the substance is dissolved in 15 c. c. of HCl, filtered and residue washed thoroughly.‡

The solution is made up to 250 c. c. in a beaker and boiled. To the

*The volume of wash-water should not be too large; vide Hillebrand.

†The accuracy of this method admits of criticism, but its convenience and rapidity demand its insertion.

‡Evaporation to dryness is unnecessary, unless gelatinous silica should have separated and should never be performed on a bath heated by gas; vide Hillebrand.

boiling solution 10 c. c. of a saturated solution of $BaCl_2$ is added slowly drop by drop from a pipette and the boiling continued until the precipitate is well formed, or digestion on the steam bath, may be substituted for the boiling. It is then set aside over night, or for a few hours, filtered, ignited and weighed as $BaSO_4$.

Total Sulphur.

One gram of the material is weighed out in a large platinum crucible and fused with $NaCO_3$ and a little KNO_3, being careful to avoid contamination from sulphur in the gases from source of heat. This may be done by fitting the crucible in a hole in an asbestos board. The melt is treated in the crucible with boiling water and the liquid poured into a tall narrow beaker and more hot water added until the mass is disintegrated. The solution is then filtered. The filtrate contained in a No. 4 beaker is to be acidulated with HCl and made up to 250 c. c. with distilled water, boiled, the sulphur precipitated as $BaSO_4$, and allowed to stand over night or for a few hours.

Loss on Ignition.

Half a gram of cement is to be weighed out in a platinum crucible, placed in a hole in an asbestos board so that about 3-5 of the crucible projects below, and blasted 15 minutes, preferably with an inclined flame. The loss by weight, which is checked by a second blasting of 5 minutes, is the loss on ignition.

May, 1903: Recent investigations have shown that large errors in results are often due to the use of impure distilled water and reagents. The analyst should, therefore, test his distilled water by evaporation and his reagents by appropriate tests before proceeding with his work.

APPENDIX C.

Standard Cement Specifications of the American Society for Testing Materials.

Adopted November 14th, 1904.

General Observations.

1.—These remarks have been prepared with a view of pointing out the pertinent features of the various requirements and the precautions to be observed in the interpretation of the results of the tests.

2.—The Committee would suggest that the acceptance or rejection under these specifications be based on tests made by an experienced person having the proper means for making the tests.

Specific Gravity.

3.—Specific gravity is useful in detecting adulteration or under-burning. The results of tests of specific gravity are not necessarily conclusive as an indication of the quality of a cement, but when in combination with the results of other tests may afford valuable indications.

Fineness.

4.—The sieves should be kept thoroughly dry.

Time of Setting.

5.—Great care should be exercised to maintain the test pieces under as uniform conditions as possible. A sudden change or wide range of temperature in the room in which the tests are made, a very dry or humid atmosphere, and other irregularities vitally affect the rate of setting.

Tensile Strength.

6.—Each consumer must fix the minimum requirements for tensile strength to suit his own conditions. They shall, however, be within the limits stated.

Constancy of Volume.

7.—The tests for constancy of volume are divided into two classes, the first normal, the second accelerated. The latter should be regarded as a precautionary test only, and not infallible. So many conditions enter into the making and interpreting of it that it should be used with extreme care.

8.—In making the pats the greatest care should be exercised to avoid initial strains due to moulding or to too rapid drying-out during the first twenty-four hours. The pats should be preserved under the most uniform conditions possible, and rapid changes of temperature should be avoided.

9.—The failure to meet the requirements of the accelerated tests need not be sufficient cause for rejection. The cement may, however, be held for twenty-eight days, and a retest made at the end of that period. Failure to meet the requirements at this time should be considered sufficient cause for rejection, although in the present state of our knowledge it cannot be said that such failure necessarily indicates unsoundness, nor can the cement be considered entirely satisfactory simply because it passes the tests.

General Conditions.

1.—All cement shall be inspected.

2.—Cement may be inspected either at the place of manufacture or on the work.

3.—In order to allow ample time for inspecting and testing, the cement should be stored in a suitable weather-tight building having the floor properly blocked or raised from the ground.

4.—The cement shall be stored in such a manner as to permit easy access for proper inspection and identification of each shipment.

5.—Every facility shall be provided by the contractor and a period of at least twelve days allowed for the inspection and necessary tests.

6.—Cement shall be delivered in suitable packages with the brand and name of manufacturer plainly marked thereon.

7. A bag of cement shall contain 94 pounds of cement net. Each barrel of Portland cement shall contain 4 bags, and each barrel of natural cement shall contain 3 bags of the above net weight.

8.—Cement failing to meet the seven-day requirements may be held awaiting the results of the twenty-eight day tests before rejection.

9.—All tests shall be made in accordance with the methods proposed by the Committee on Uniform Tests of Cement of the American Society of Civil Engineers, presented to the Society January 21, 1903, and amended January 20, 1904, with all subsequent amendments thereto.

10. The acceptance or rejection shall be based on the following requirements:

Natural Cement.

11. Definition. This term shall be applied to the finely pulverized product resulting from the calcination of an argillaceous limestone at a temperature only sufficient to drive off the carbonic acid gas.

Specific Gravity.

12.—The specific gravity of the cement thoroughly dried at 100° Cent., shall be not less than 2.8.

Fineness.

13.—It shall leave by weight a residue of not more than 10% on the No. 100, and 30% on the No. 200 sieve.

Time of Setting.

14.—It shall develop initial set in not less than ten minutes, and hard set in not less than thirty minutes, nor more than three hours.

Tensile Strength.

15.—The minimum requirements for tensile strength for briquettes one inch square in cross section shall be within the following limits, and shall show no retrogression in strength within the periods specified:*

Age. Neat Cement.	Strength.
24 hours in moist air	50-100 lbs.
7 days (1 day in moist air, 6 days in water)	100-200 "
28 days (1 day in moist air, 27 days in water)	200-300 "
One Part Cement, Three Parts Standard Sand.	
7 days (1 day in moist air, 6 days in water)	25- 75 "
28 days (1 day in moist air, 27 days in water)	75-150 "

Constancy of Volume.

16.—Pats of neat cement about three inches in diameter, one-half inch thick at center, tapering to a thin edge, shall be kept in moist air for a period of twenty-four hours.

(a) A pat is then kept in air at normal temperature.

(b) Another is kept in water maintained as near 70° Fahr. as practicable.

17.—These pats are observed at intervals for at least 28 days, and, to

*For example the minimum requirement for the twenty-four hour neat cement test should be some specified value within the limits of 50 and 100 pounds, and so on for each period stated.

satisfactorily pass the tests, should remain firm and hard and show no signs of distortion, checking, cracking or disintegrating.

Portland Cement.

18.—DEFINITION.—This term is applied to the finely pulverized product resulting from the calcination to incipient fusion of an intimate mixture of properly proportioned argillaceous and calcareous materials, and to which no addition greater than 3% has been made subsequent to calcination.

Specific Gravity.

19.—The specific gravity of the cement, thoroughly dried at 100° Cent., shall be not less than 3.10.

Fineness.

20.—It shall leave by weight a residue of not more than 8% on the No. 100, and not more than 25% on the No. 200 sieve.

Time of Setting.

21.—It shall develop initial set in not less than thirty minutes, but must develop hard set in not less than one hour, nor more than ten hours.

Tensile Strength.

22.—The minimum requirements for tensile strength for briquettes one inch square in section shall be within the following limits, and shall show no retrogression in strength within the periods specified.*

Age.	Neat Cement.	Strength.
24 hours in moist air		150-200 lbs.
7 days (1 day in moist air, 6 days in water)		450-550 "
28 days (1 day in moist air, 27 days in water)		550-650 "
	One Part Cement, Three Parts Sand.	
7 days (1 day in moist air, 6 days in water)		150-200 "
28 days (1 day in moist air, 27 days in water)		200-300 "

Constancy of Volume.

23.—Pats of neat cement about three inches in diameter, one-half inch thick at the centre, and tapering to a thin edge, shall be kept in moist air for a period of twenty-four hours.

(a) A pat is then kept in air at normal temperature and observed at intervals for at least 28 days.

(b) Another pat is kept in water maintained as near 70° Fahr. as practicable, and observed at intervals for at least 28 days.

(c) A third pat is exposed in any convenient way in an atmosphere of steam, above boiling water, in a loosely closed vessel for five hours.

24.—These pats, to satisfactorily pass the requirements, shall remain firm and hard and show no signs of distortion, checking, cracking or disintegrating.

Sulphuric Acid and Magnesia.

25.—The cement shall not contain more than 1.75% of anhydrous sulphuric acid (SO_3), nor more than 4% of magnesia (MgO).

*For example the minimum requirement for the twenty-four hour neat cement test should be some specified value within the limits of 150 and 200 pounds, and so on for each period stated.

APPENDIX D.

U. S. Army Standard Specifications.*

Recommended by the Board of Engineer Officers on Testing Hydraulic Cements, for use in the Engineer Department, U. S. Army. Major William L. Marshall, Major Smith S. Leach, Captain Spencer Cosby, Corps of Engineers, Members of Board.

Specifications for American Portland Cement.

(1) The cement shall be an American Portland, dry, and free from lumps. By a Portland cement is meant the product obtained from the heating or calcining up to incipient fusion of intimate mixtures, either natural or artificial, of argillaceous with calcareous substances, the calcined product to contain at least 1.7 times as much of lime, by weight, as of the materials which give the lime its hydraulic properties, and to be finely pulverized after said calcination, and thereafter additions or substitutions for the purpose only of regulating certain properties of technical importance to be allowable to not exceeding 2 per cent. of the calcined product.

(2) The cement shall be put up in strong, sound barrels well lined with paper, so as to be reasonably protected against moisture, or in stout cloth or canvas sacks. Each package shall be plainly labeled with the name of the brand and of the manufacturer. Any package broken or containing damaged cement may be rejected or accepted as a fractional package, at the option of the United States agent in local charge.

(3) Bidders will state the brand of cement which they propose to furnish. The right is reserved to reject a tender for any brand which has not established itself as a high-grade Portland cement and has not for three years or more given satisfaction in use under climatic or other conditions of exposure of at least equal severity to those of the work proposed.

(4) Tenders will be received only from manufacturers or their authorized agents.

(The following paragraph will be substituted for paragraphs 3 and 4 above when cement is to be furnished and placed by the contractor:

No cement will be allowed to be used except established brands of high-grade Portland cement which have been made by the same mill and in successful use under similar climatic conditions to those of the proposed work for at least three years.)

(5) The average weight per barrel shall not be less than 375 pounds net. Four sacks shall contain one barrel of cement. If the weight, as determined by test weighings, is found to be below 375 pounds per barrel, the cement may be rejected, or, at the option of the engineer officer in charge, the contractor may be required to supply, free of cost to the United States, an additional amount of cement equal to the shortage.

(6) Tests may be made of the fineness, specific gravity, soundness, time of setting, and tensile strength of the cement.

*Professional Papers No. 28, Corps of Engineers, U. S. Army.

(7) Fineness.—Ninety-two per cent. of the cement must pass through a sieve made of No. 40 wire, Stubb's gauge, having 10,000 openings per square inch.

(8) Specific Gravity.—The specific gravity of the cement as determined from a sample which has been carefully dried, shall be between 3.10 and 3.25.

(9) Soundness.—To test the soundness of the cement, at least two pats of neat cement mixed for five minutes with 20 per cent. of water by weight shall be made on glass, each pat about 3 inches in diameter and one-half inch thick at the center, tapering thence to a thin edge. The pats are to be kept under a wet cloth until finally set, when one is to be placed in fresh water for twenty-eight days. The second pat will be placed in water which will be raised to the boiling point for six hours, then allowed to cool. Neither should show distortion or cracks. The boiling test may or may not reject at the option of the engineer officer in charge.

(10) Time of Setting.—The cement shall not acquire its initial set in less than forty-five minutes and must have acquired its final set in ten hours.

(The following paragraph will be substituted for the above in case a quick-setting cement is desired:

The cement shall not acquire its initial set in less than twenty nor more than thirty minutes, and must have acquired its final set in not less than forty-five minutes nor more than two and one-half hours.)

The pats made to test the soundness may be used in determining the time of setting. The cement is considered to have acquired its initial set when the pat will bear, without being appreciably indented, a wire one-twelfth inch in diameter loaded to weigh one-fourth pound. The final set has been acquired when the pat will bear, without being appreciably indented, a wire one-twenty-fourth inch in diameter loaded to weigh 1 pound.

(11) Tensile Strength.—Briquettes made of neat cement, after being kept in air for twenty-four hours under a wet cloth and the balance of the time in water, shall develop tensile strength per square inch as follows:

After seven days, 450 pounds; after twenty-eight days, 540 pounds.

Briquettes made of 1 part cement and 3 parts standard sand, by weight, shall develop tensile strength per square inch as follows:

After seven days, 140 pounds; after twenty-eight days, 220 pounds.

(In case quick-setting cement is desired, the following tensile strengths shall be substituted for the above:

Neat briquettes: After seven days, 400 pounds; after twenty-eight days, 480 pounds.

Briquettes of 1 part cement to 3 parts standard sand: After seven days, 120 pounds; after twenty-eight days, 180 pounds.)

(12) The highest result from each set of briquettes made at any one time is to be considered the governing test. Any cement not showing an increase of strength in the twenty-eight day tests over the seven-day tests will be rejected.

(13) When making briquettes neat cement will be mixed with 20 per cent. of water by weight, and sand and cement with 12½ per cent. of water by weight. After being thoroughly mixed and worked for five minutes, the cement or mortar will be placed in the briquette mold in four equal layers, and each layer rammed and compressed by thirty blows of a soft brass or copper rammer three-quarters of an inch in diameter (or seven-tenths of an inch square, with rounded corners), weighing 1 pound. It is to be allowed to drop on the mixture from a height of about half an inch. When the ramming has been completed the surplus cement shall be struck off and the final layer smoothed with a trowel held almost horizontal and drawn back with sufficient pressure to make its edge follow the surface of the mold.

(14) The above are to be considered the minimum requirements. Unless a cement has been recently used on work under this office, bidders will deliver a sample barrel for test before the opening of bids. If this sample shows higher tests than those given above, the average of tests made on subsequent shipments must come up to those found with the sample.

(15) A cement may be rejected in case it fails to meet any of the above requirements. An agent of the contractor may be present at the making of the tests, or, in case of the failure of any of them, they may be repeated in his presence. If the contractor so desires, the engineer officer in charge may, if he deem it to the interest of the United States, have any laboratory in the manner herein specified. All expenses of such tests to be or all of the tests made or repeated at some recognized standard testing paid by the contractor. All such tests shall be made on samples furnished by the engineer officer from cement actually delivered to him.

Specifications for Natural Cement.

(1) The cement shall be a freshly packed natural or Rosendale, dry, and free from lumps. By Natural cement is meant one made by calcining natural rock at a heat below incipient fusion, and grinding the product to powder.

(2) Same as Portland (2).

(3) Bidders will state the brand of cement which they propose to furnish. The right is reserved to reject a tender for any brand which has not given satisfaction in use under climatic or other conditions of exposure of at least equal severity to those of the work proposed.

(4) Tenders will be received only from manufacturers or their authorized agents.

(The following paragraph will be substituted for paragraphs 3 and 4 above when cement is to be furnished and placed by the contractor:

No cement will be allowed to be used except established brands of high-grade natural cement which have been in successful use under similar climatic conditions to those of the proposed work.)

(5) The average net weight per barrel shall not be less than 300 pounds. (West of the Allegheny Mountains this may be 265 pounds.) . . . sacks of cement shall have the same weight as 1 barrel. If the average

net weight, as determined by test weighings, is found to be below 300 pounds (265 pounds) per barrel, the cement may be rejected, or, at the option of the engineer officer in charge, the contractor may be required to supply free of cost to the United States an additional amount of cement equal to the shortage.

(6) Tests may be made of the fineness, time of setting, and tensile strength of the cement.

(7) Fineness.—At least 80 per cent. of the cement must pass through a sieve made of No. 40 wire, Stubb's gauge, having 10.000 openings per square inch.

(8) Time of Setting.—The cement shall not acquire its initial set in less than twenty minutes and must have acquired its final set in four hours.

(9) The time of setting is to be determined from a pat of neat cement mixed for five minutes with 30 per cent. of water by weight and kept under a wet cloth until finally set. The cement is considered to have acquired its initial set when the pat will bear, without being appreciably indented, a wire one-twelfth inch in diameter loaded to weigh one-fourth pound. The final set has been acquired when the pat will bear, without being appreciably indented, a wire one-twenty-fourth inch in diameter loaded to weigh 1 pound.

(10) Tensile Strength.—Briquettes made of neat cement shall develop the following tensile strengths per square inch after having been kept in air for twenty-four hours under a wet cloth and the balance of the time in water:

At the end of seven days, 90 pounds; at the end of twenty-eight days, 200 pounds.

Briquettes made of one part cement and one part standard sand by weight shall develop the following tensile strengths per square inch:

After seven days, 60 pounds; after twenty-eight days, 150 pounds.

(11) Same as Portland (12).

(12) Same as Portland (13) except that 30 and 17 per cent. of water are used for neat and sand mixtures respectively.

(13) Same as Portland (14).

(14) Same as Portland (15).

Specifications for Puzzolan Cement.

(1) The cement shall be a Puzzolan of uniform quality, finely and freshly ground, dry, and free from lumps, made by grinding together without subsequent calcination granulated blast-furnace slag with slaked lime.

(2) Same as Portland (2).

(3) Bidders will state the brand of cement which they propose to furnish. The right is reserved to reject a tender for any brand which has not given satisfaction in use under climatic or other conditions of exposure of at least equal severity to those of the work proposed, and for any brand from cement works that do not make and test the slag used in the cement.

(4) Tenders will be received only from manufacturers or their authorized agents.

(The following paragraph will be substituted for paragraphs 3 and 4 above when cement is to be furnished and placed by the contractor:

No cement will be allowed to be used except established brands of high-grade Puzzolan cement which have been in successful use under similar climatic conditions to those of the proposed work and which come from cement works that make the slag used in the cement.)

(5) The average weight per barrel shall not be less than 330 pounds net. Four sacks shall contain 1 barrel of cement. If the weight as determined by test weighings is found to be below 330 pounds per barrel, the cement may be rejected or, at the option of the engineer officer in charge, the contractor may be required to supply, free of cost to the United States, an additional amount of cement equal to the shortage.

(6) Tests may be made of the fineness, specific gravity, soundness, time of setting, and tensile strength of the cement.

(7) Fineness.—Ninety-seven per cent. of the cement must pass through a sieve made of No. 40 wire, Stubb's gauge, having 10,000 openings per square inch.

(8) Specific Gravity.—The specific gravity of the cement as determined from a sample which has been carefully dried, shall be between 2.7 and 2.8.

(9) Soundness.—To test the soundness of cement, pats of neat cement mixed for five minutes with 18 per cent. of water by weight shall be made on glass, each pat about 3 inches in diameter and one-half inch thick at the center, tapering thence to a thin edge. The pats are to be kept under wet cloths until finally set, when they are to be placed in fresh water. They should not show distortion or cracks at the end of twenty-eight days.

(10) Time of Setting.—The cement shall not acquire its initial set in less than forty-five minutes and shall acquire its final set in ten hours. The pats made to test the soundness may be used in determining the time of setting. The cement is considered to have acquired its initial set when the pat will bear, without being appreciably indented, a wire one-twelfth inch in diameter loaded to one-fourth pound weight. The final set has been acquired when the pat will bear, without being appreciably indented, a wire one twenty-fourth inch in diameter loaded to 1 pound weight.

(11) Tensile Strength.—Briquettes made of neat cement, after being kept in air under a wet cloth for twenty-four hours and the balance of the time in water, shall develop tensile strength per square inch as follows:

After seven days, 350 pounds; after twenty-eight days, 500 pounds.

Briquettes made of one part cement and three parts standard sand by weight shall develop tensile strength per square inch as follows:

After seven days, 140 pounds; after twenty-eight days, 220 pounds.

(12) Same as Portland (12).

(13) Same as Portland (13) except that 18 and 10 per cent. of water are used for neat and sand mixtures respectively.

(14) Same as Portland (14).

(15) Same as Portland (15).

APPENDIX E.

British Standard Specifications for Portland Cement.

Issued by the Engineering Standards Committee, supported by the Institution of Civil Engineers, The Institution of Mechanical Engineers, the Institution of Naval Architects, the Iron and Steel Institute and the Institution of Electrical Engineers.

Quality and Preparation.

1.—The cement is to be prepared by intimately mixing together calcareous and argillaceous materials, burning them at a clinkering temperature and grinding the resulting clinker. No addition of any material is to be made after burning, except when desired by the manufacturer, and if not prohibited in writing by the consumer, in which case calcium sulphate or water may be used. The cement, if watered, shall contain not more than 2 per cent. of water, whether that water has been added or has been naturally absorbed from the air. If calcium sulphate is used, not more than 2 per cent. calculated as anhydrous calcium sulphate of the weight of the cement shall be added.

Sampling and Preparation for Testing and Analysis.

2.—As soon as the cement has been bulked at the makers' works,* or on the works in connection with which the material is to be used, at the consumer's option, samples for testing are to be taken from each parcel, each sample consisting of cement from at least twelve different positions in the same heap, so distributed as to ensure, as far as is practicable, a fair average sample of the whole parcel, all to be mixed together and the sample for testing to be taken therefrom.

3.—Before gauging the tests, the sample so obtained is to be spread out for a depth of 3 ins. for 24 hours, in a temperature of 58 to 64 degrees Fahrenheit.

4.—In all cases where consignments are of 100 tons and upwards, samples selected as above from each consignment, either at the makers' works or after delivery at the works where the cement is to be used, are to be sent for expert testing and for chemical analysis. In no case is cement so tested and analyzed to be accepted, or used, unless previously certified in writing by the consumer to be of satisfactory quality. Payment for such tests and analysis to be made by the consumer, the manufacturer supplying the cement required for the same, free of charge.

When consignments of less than 100 tons have to be supplied, the makers shall, if required, give certificates for each delivery, to the effect that such cement complies with the terms of this standard specification, with regard to quality, tests, and chemical analysis, no payment being made by the consumer for such certificate, nor for the making of such tests and analyses.

5.—Should it be deemed more convenient by the consumers, that the

*Should the consumer desire to stipulate for any special quantity, the size of the heap should be stated.

samples for testing should be taken at the maker's works before delivery, the latter are, in that event, to afford full facilities to the inspector, who may be appointed by the consumers to sample the cement as he may desire at the maker's works, and subsequently to identify each parcel as it may be despatched, with that sampled by him. No parcel is to be sent away, unless a written order has been previously received by the makers from the said consumer to the effect that the material in question has been approved.

Fineness and Sieves.

6.—The cement shall be ground to comply with the following degrees of fineness, viz:

The residue on a sieve 76 × 76 = 5,776 meshes per square inch is not to exceed 5 per cent.

The residue on a sieve 180 × 180 = 32,400 meshes per square inch is not to exceed 22½ per cent.

The sieves are to be prepared from standard wire, the size of the wire for the 5,776 mesh is to be .0044 inch, and for the 32,400 mesh, .0018 inch. The wire shall be woven (not twilled), the cloth being carefully mounted on the frames without distortion.

Specific Gravity.

7.—The specific gravity of the cement shall be not less than 3.15 when sampled and hermetically sealed at the maker's works, nor less than 3.10 if sampled after delivery to the consumer.

Chemical Composition.

8.—The cement is to comply with the following conditions as to its chemical composition. There shall be no excess of lime, that is to say, the proportion of lime shall be not greater than is necessary to saturate the silica and alumina present. The percentage of insoluble residue shall not exceed 1.5 per cent.; that of magnesia shall not exceed 3 per cent., and that of sulphuric anhydride shall not exceed 2.5 per cent.

Tensile Tests.

9.—The quantity of water used in gauging shall be appropriate to the quality of the cement, and shall be so proportioned that when the cement is gauged it shall form a smooth, easily worked paste, that will leave the trowel cleanly in a compact mass. Fresh water is to be used for gauging, the temperature thereof, and of the test room at the time the said operations are performed, being from 58 to 64 degrees Fahrenheit.

The cement gauged as above is to be filled, without mechanical ramming, into moulds of the form shown in Fig. 140 on the annexed sketch, each mould resting upon an iron plate until the cement has set. When the cement has set sufficiently to enable the mould to be removed without injury to the briquette, such removal is to be effected. The said briquette shall be kept in a damp atmosphere and placed in fresh water 24 hours after gauging, and kept there until broken, the water in which the test briquettes are submerged being renewed every seven days, and the temperature thereof maintained between 58 and 64 degrees Fahrenheit.

Neat Tests.

10.—Briquettes of neat cement of the shape shown in Fig. 140 and annexed thereto are to be gauged for breaking at 7 and 28 days, respectively, six briquettes for each period. The average tensile strength of the six briquettes shall be taken as the accepted tensile strength for each

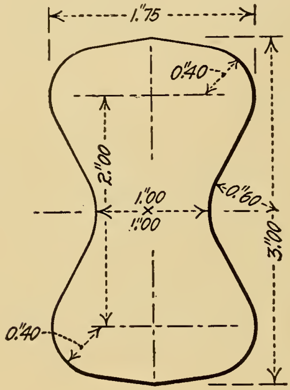


Fig. A.
Dimensions of Briquette.

FIG. 140.

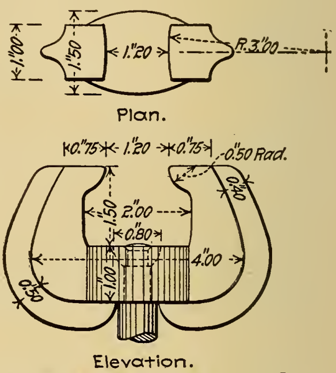


Fig. B, Details of Jaws for Holding Briquette.

FIG. 141.

period. For breaking, the briquette is to be held in strong metal jaws, of the shape shown in Fig. 141 on the annexed sketch, the briquettes being slightly greased where gripped by the jaws. The load must then be steadily and uniformly applied, starting from zero, increasing at the rate of 100 pounds in 12 seconds. The briquettes are to bear on the average not less than the following tensile stresses before breaking:

7 days from gauging..............400 lbs. per sq. inch of section.
28 days from gauging..............500 lbs. per sq. inch of section.
The increase from 7 to 28 days shall not be less than:
25% when the 7-day test falls between 400 lbs. to 450 lbs. per sq. in.
20% when the 7-day test falls between 450 lbs. to 500 lbs. per sq. in.
15% when the 7-day test falls between 500 lbs. to 550 lbs. per sq. in.
10% when the 7-day test falls between 550 lbs. per sq. in. or upwards.

Sand Tests.

11.—The cement shall also be tested by means of briquettes prepared from one part of cement to three parts of weight of dry standard sand, the said briquettes being of the shape described for the neat cement tests, the mode of gauging, filling the moulds, and breaking the briquettes is also to be similar. The proportion of water used shall be such that the mixture is thoroughly wetted, and there shall be no superfluous water when the briquettes are formed. The cement and sand briquettes are to bear the following tensile stresses:

7 days from gauging..............120 lbs. per sq. inch of section.
28 days from gauging..............225 lbs. per sq. inch of section.
The increase from 7 to 28 days shall not be less than 20 per cent.

The standard sand referred to above is to be obtained from Leighton Buzzard. It must be thoroughly washed, dried and passed through a sieve of 20 × 20 meshes per square inch, and must be retained on a sieve of 30 × 30 meshes per square inch, the wires of the sieve being .0164 and .0108 inch, respectively.

Setting Time.

12.—There shall be three distinct gradations of setting time, which shall be designated as "quick," "medium," and "slow."*

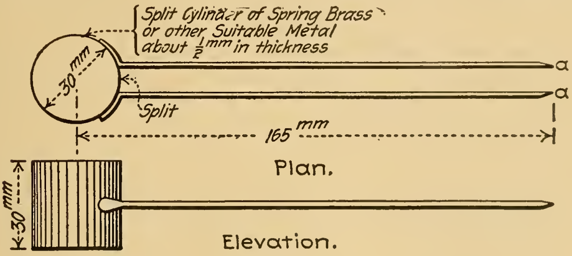
Quick—The setting time shall be not less than ten minutes, or more than 30 minutes.

Medium—The setting time shall be not less than half an hour, or more than two hours.

Slow—The setting time shall be not less than two hours, or more than five hours.*

The temperature of the air in the test room at the time of gauging and of the water used is to be between 58 and 64 degrees Fahrenheit.

The cement shall be considered as "set" when a needle having a flat



Apparatus for Conducting the "Le Chatelier" Test.

FIG. 142.

end 1-16 inch square, weighing in all 2½ lbs., fails to make an impression when its point is applied gently to the surface.

Soundness.

13.—The cement shall be tested by the Le Chatelier method, and is in no case to show a greater expansion than 12 millimetres after 24 hours' aeration and 6 millimetres after seven days' aeration.

NOTE.—The apparatus for conducting the Le Chatelier test (Fig. 142) consists of a small split cylinder of spring brass or other suitable metal of 0.5 millimetre (0.197 inch) in thickness, 30 millimetres (1.1875 inches) internal diameter, and 30 millimetres high, forming the mould, to which on either side of the split are attached two indicators 165 millimetres (6.5 inches) long from the center of the cylinder, with pointed ends A A, as shown upon the sketch.

In conducting the test, the mould is to be placed upon a small piece of glass and filled with cement gauged in the usual way, care being taken to keep the edges of the moulds gently together while this operation is

*When a specially slow setting cement is required, the minimum time of setting shall be specified.

being performed. The mould is then covered with another glass plate, a small weight is placed on this and the mould is immediately placed in water at 58 to 64 degrees Fahrenheit, and left there for 24 hours.

The distance separating the indicator points is then measured, and the mould placed in cold water, which is brought to a boiling point in 15 to 30 minutes, and kept boiling for six hours. After cooling, the distance between the points is again measured; the difference between the two measurements represents the expansion of the cement, which must not exceed the limits laid down in this specification.

14.—The tests and analyses hereinbefore referred to shall in no case relate to a larger quantity of cement than 250 tons sampled at one time.

Acceptance.

15.—No cement is to be approved or accepted unless it fully complies with the foregoing conditions.

APPENDIX F.

Standard Specifications for Portland Cement.

Adopted by the Canadian Society of Civil Engineers.
Report submitted, January 27th, 1903.

Committee:—Dr. H. T. Bovey (chairman), Messrs. M. J. Butler, C. B. Smith, T. Monro, P. A. Peterson, C. H. Rust, G. A. Mountain and J. A. Duff

The whole of the cement is to be well-burned, pure Portland cement, of the best quality, free from free-lime, slag, dust, or other foreign material.

(1) Fineness.—The cement shall be ground so fine that the residue on a sieve of 10,000 meshes to the square inch shall not exceed ten per cent. of the whole by weight, and the whole of the cement shall pass a sieve of 2,500 meshes to the square inch.

(2) Specific Gravity.—The specific gravity of the cement shall be at least 3.09 and shall not exceed 3.25 for fresh cement; the term "fresh" being understood to apply to such cements as are not more than two months old.

(3) Tests.—The cement shall be subjected to the following tests:

(a) Blowing Test.—Mortar pats of neat cement thoroughly worked, shall be trowelled upon, carefully cleaned, 5 in. by 2½-in. ground glass plates. The pats shall be about ½ in. thick in the center and worked off to the sharp edges at the four sides. They shall be covered with a damp cloth and allowed to remain in the air until set, after which they shall be placed in vapor in a tank, in which the water is heated to a temperature of 130° Fahr. After remaining in the vapor six hours, including the time of setting in air, they shall be immersed in the hot water and allowed to remain there for eighteen hours. After removal from the water the samples shall not be curled up, shall not have fine hair cracks, nor large expansion cracks, nor shall they be distorted. If separated from the glass, the samples shall break with a sharp, crisp ring.

(b) Tensile Test. (Neat Cement.)—Briquettes made of neat cement, mixed with about twenty per cent. of water, by weight, after remaining one day in air, in a moist atmosphere, shall be immersed in water, and shall be capable of sustaining a tensile stress of 250 lbs. per square inch after submersion for two days; 400 lbs. per square inch after submersion for six days; 500 lbs. per square inch after submersion for twenty-seven days. The tensile test shall be considered as the average of the strength of five briquettes, and any cement showing a decrease in tensile strength on or before the twenty-eighth day shall be rejected.

(Sand and Cement).—The sand for standard tests shall be clean quartz, crushed so that the whole shall pass through a sieve of 400 meshes to the square inch, but shall be retained on a sieve of 900 meshes per square inch. The sand and cement shall be thoroughly mixed dry, and then about ten per cent. of their weight of water shall be added, when the briquettes are to be formed in suitable moulds. After remaining in a damp chamber for twenty-four hours the briquettes shall be immersed in water, and briquettes made in the proportion of one of cement to three of sand, by weight, shall bear a tensile stress of 125 lbs. per square inch after submersion for six days, and 200 lbs. per square inch after submersion for twenty-eight days. Sand and cement briquettes shall not show a decrease in tensile strength at the end of twenty-eight days, or subsequently.

(4) The manufacturer shall, if required, supply chemical analyses of the cement.

(5) Packing.—The cement shall be packed either in stout air and water-tight casks, carefully lined with strong brown paper, or in strong air and water-tight bags.

(6) The manufacturer shall give a certificate with each shipment of cement stating (1) the date of manufacture; (2) the tests and analyses which have been obtained for the cement in question at the manufacturer's laboratory; (3) that the cement does not contain any adulterations.

(Note: This specification is followed by a standard method of testing.)

APPENDIX G

Bibliography.

A brief list of the books, in the English language, on cement and concretes, best suited for reference, with the essential features of each.

Bleninger., A. V., The Manufacture of Hydraulic Cements. Fourth Series,

Bulletin No. 3, State Geological Survey of Ohio, 1904.
(Chemistry of cements and cement materials, and methods of manufacture.)

Brown, Charles C. Directory of American Cement Industries and Hand Book for Cement Users. Municipal Engineering Co., Indianapolis, Ind.—Published yearly.
(Directory of brands, manufacturers, agents, users, with other data on the industry.)

Buel, A. W. and C. S. Hill. Reinforced Concrete. Engineering News Publishing Co., New York, 1904.
(Mathematical discussion, and practical consideration of constructions of reinforced concrete.)

Burr, William H. The Elasticity and Resistance of the Materials of Engineering. John Wilev & Sons, New York, 1903.
(Chiefly mathematical, little practical data.)

Butler, David B. Portland Cement, Its Manufacture, Testing and Use. Spon, London, 1899.
(English practice in manufacture and testing.)

Cement Industry, The. The Engineering Record, New York, 1900.
(Descriptions of plants and processes.)

Considère, A. Experimental Researches on Reinforced Concrete, translated and arranged by Leon S. Moisseiff. McGraw Publishing Co., New York, 1903.
(Experiments and data on reinforced concrete.)

Cummings, Uriah. American Cements. Rogers and Manson, Boston, 1898.
(Historical data, and discussion of natural cements.)

Eckel, Edwin C. Cements, Limes, and Plasters. John Wiley & Sons, New York, 1905.
(Cement materials and processes of manufacture.)

Falk, Myron S. Cements, Mortars and Concretes. Myron C. Clark, New York, 1904.
(Data on investigations of physical properties.)

Gillette, Halbert P. Hand-book of Cost Data. Myron C. Clark, New York, 1905. (Costs of Concrete and Concrete-Steel Structures.)

Grant, John. Portland Cement; Its Nature, Tests, and Uses. Spon, London, 1875.
(Data on strength of cement—chiefly of historic interest.)

Johnson, J. B. The Materials of Construction. John Wiley & Sons, New York, 1898.
(Mathematical discussion, general description, and much valuable data.)

Le Chatelier, H. Experimental Researches on the Constitution of Hydraulic Mortars, translated by Joseph L. Mack. McGraw Publishing Co., New York, 1905.
(Chemical and mineralogical study of cement composition.)

Marsh, Charles F. Reinforced Concrete. D. Van Nostrand Co., New York, 1904.
(Theoretical and practical discussion of concrete structures.)

Meade, Richard K. The Examination of Portland Cements. The Chemical Publishing Co., Easton, Pa., 1901.
(Methods of chemical analysis.)

Redgrave, Gilbert R. and C. Spackman. Calcareous Cements: Their Nature and Use. Chas. Griffin & Co., London, 1905.
(Historical data, and English methods of manufacture and testing.)

Sabin, Louis C. Cement and Concrete. McGraw Publishing Co., New York, 1905.
(Valuable data on the properties of cement, and their application to practical construction.)

Spalding, Frederick P. Hydraulic Cement: Its Properties, Testing and Use. John Wiley & Sons, New York, 1897.
(The nature and testing of cement—no data.)

Taylor, Frederick W. and Sanford E. Thompson. A Treatise on Concrete, Plain and Reinforced. John Wiley & Sons, New York, 1904.
(Data on cements and concretes, and practical application to construction work.)

INDEX