A MANUAL OF
CEMENT TESTING

FOR THE USE OF ENGINEERS AND CHEMISTS IN COLLEGES AND IN THE FIELD

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PREFACE.

In order to insure uniformity of results in the testing of cement, it is essential that each test should invariably be made in precisely the same manner and under exactly the same conditions. A committee of the American Society of Civil Engineers has, with this aim in view, prepared and published a set of Standard Methods of Testing Cement, and these methods are to-day employed throughout the United States.

This little volume, as its name implies, is a laboratory manual on cement testing, and is intended to assist in bringing about uniformity in the testing of cement. The authors have endeavored to present, in a somewhat condensed form, such directions as will enable a student in the laboratory or an operator in the field office to correctly interpret the Standard Methods of Testing and Specifications for Cement as published by the committee of the American Society of Civil Engineers, American Society for Testing Materials, Association of American Portland Cement Manufacturers, and the American Railway Engineers and Maintenance of Way Association; they have endeavored to give sufficient detail to enable all students to learn the same manipulations and thus be able to perform each test in a certain well defined and similar manner.

All of the tests described have been performed in the laboratory under the eyes of the writers and have been found to produce uniformly good results.

Acknowledgment has been made to various authorities on the subject by special mention or as references at the end of the several chapters.

Any corrections or suggestions will be gratefully received.

W. A. R.
H. B. N.

June 15th, 1912.

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PART I.

INTRODUCTION.

THE Portland Cement Industry in the United States has had a most marvelous development. In 1880 the United States produced 82,000 barrels of Portland Cement, while in 1910 the output was estimated to be 70,000,000 barrels. This great increase in the use of cement is fitting testimony of its great value as a material of construction.

Like all other materials used in construction, cement must be tested. Iron, steel, wood and stone, in their preparation for use or tests, have only their shapes changed; but it is quite different with cement, which comes from the manufacturer to the testing laboratory, or wherever it is to be used, in the form of a fine powder, there to be mixed with water into a paste and deposited in forms till hardened into a solid. It is evident that of all materials of construction subjected to a system of testing, cement is probably the most dependent on the judgment and skill of the person making the tests. It was with a view to eliminating this personal factor as far as possible, and thereby placing the tests of one operator, or one laboratory, on a basis of comparison with another operator or laboratory, that the American Society of Civil Engineers and several other societies appointed a committee to draw up specifications to be used in all tests of cement in the United States.

These specifications form the basis of this manual, and whenever the term "Standard Specifications," is used, it refers to the "Standard Methods of Testing and Specifications for Cement," of the American Society of Civil Engineers.

Classes. Cement tests are of two classes: (1) Experimental Tests, made for scientific purposes, and comprising such tests as modulus of elasticity, coefficient of expansion, etc., and (2) Routine Tests, made to ascertain if a certain consignment of cement will answer the requirements of a set of specifications, as regards soundness and strength.

The routine tests usually employed are fineness, specific gravity, soundness, tensile strength (both neat and sand), and time of set, while compression and transverse tests are sometimes used.

The tests for soundness and strength are called primary tests, while fineness, specific gravity, etc., are called secondary, since they give only additional information, which is of little value in itself.

Routine tests alone will be considered in this work.

CEMENT TESTING

CHAPTER I.

CLASSIFICATION, COMPOSITION, MANUFACTURE.

Definition. Hydraulic cement is a material, which, when pulverized and mixed into a more or less pasty mass with water, has the property of setting or hardening under water.

Classification. Cements are usually classified as follows: (1) Portland, (2) natural, (3) Pozzuolana, (4) blended or mixed.

Portland cement is the finely ground powder of a clinker resulting from the incipient fusion of an intimate artificial mixture of finely ground calcareous and argillaceous* materials, and must contain no materials added after calcination other than a small amount of calcium sulphate to regulate setting.†

Natural cement is the finely ground powder of a clinker,

* Calcareous — partaking of the nature of calcite or calcium carbonate Argillaceous — of a clayey nature.

† This definition is sometimes further limited by stating: The finished product must contain at least 1.7 times as much lime, by weight, as silica alumina and iron oxide combined.

resulting from the burning, at a heat below incipient fusion, of argillaceous limestone or other suitable natural rock.

Pozzuolana cement results from grinding and mixing in definite proportions slaked lime and blast furnace slag, or certain volcanic lava.

Mixed cement is, as the name implies, a cement made up of different brands or kinds of cements, and sometimes inert substances.

Distinguishing Features. Natural or common cements are light or dark gray, according to the stone from which they are made. The specific gravity is from 2.7 to 3.0, with an average about 2.85. Portland cements have a specific gravity of from 3.0 to 3.5, averaging about 3.15. Natural cements have much quicker set and are lower in strength in the earlier tests.

Pozzuolana cement made from slag is characterized chiefly by its light lilac color, absence of grit, low specific gravity (2.6–2.8), and by the intense bluish green color of a fresh fracture after long submersion in water.

Composition. The basic elements of Portland cement are silica, alumina and lime. Ingredients such as iron, magnesia, alkalies, sulphuric acid, carbonic acid, and water also occurs in varying quantities, replacing some of the basic elements.

The following represents about the limits within which fall the constituents of various American Portland cements, which pass the Standard Specifications for soundness, setting time and tensile strength:

	Per Cent.
Silica	20 to 24
Alumina	5 to 9
Iron Oxide	2 to 4
Lime	60 to 63.5
Magnesia	1 to 2
Sulphur Trioxide	1.5

The following represents an average:

	Per Cent.
Silica	22.0
Alumina	7.5
Iron Oxide	2.5
Lime	62.0
Magnesia	2.5
Sulphur Trioxide	1.5

Silica (SiO_2). 19–24 per cent, exists in combination with lime as calcium silicate, which is an active hardening factor. It should not be present as free silica.

Lime (CaO). 59–67 per cent, depending on the relative proportions of alumina and silica and care with which the cement has been manufactured. When in the combined state the greater the amount the stronger the cement. Excess of lime, or lime in the free state, will make an unsound cement by expanding due to slaking. The more lime, the slower the setting.

Alumina (Al_2O_3). 5–10 per cent, mostly combined as calcium aluminate. The greater the proportion, the quicker the setting and the lower ultimate tensile strength. Le Chatelier believes calcium aluminate to be the greatest factor in hardening.

Iron Oxide (Fe_2O_3). Usually less than 4 per cent. Probably has little influence on the cement, though believed by some to act the same as alumina.

Magnesia (MgO). 2–4 per cent, by some considered as an impurity, while other investigators claim it acts the same as lime. Four per cent is placed as the limit by the Standard Specifications.

Sulphuric Acid or Sulphur Trioxide (SO_3). 1.25–1.75 per cent, due mostly to the introduction of calcium sulphate into the finished cement to regulate setting. The more calcium sulphate ($CaSO_4$) the slower the set. It should never exceed 2–3 per cent, while the Standard Specifications limits it to 1.75.

Sulphur (S). Found only in small amounts, usually comes from the coal used in burning the clinker, though sometimes from the raw materials. Sulphides, when in any considerable quantity, cause discolorations (dark blue spots) in the cement on hardening, and disintegration due to oxidation.

Alkalies (K_2O and Na_2O). 0.5–2 per cent, have little or no effect on cement unless in large quantities.

Carbonic Acid (CO_2). 0.5–1.5 per cent, due mostly to absorption from the air; a large proportion shows underburning or excess of lime.

Natural and Pozzuolana Cements. The constituents of natural and Pozzuolana cements are practically the same as those of Portland cement, except that in the natural they are found in varying proportions.

The following analyses* will serve to illustrate.

* From Eckel's "Cement Materials and Industry."

	Portland.	Natural.	Pozzuolana.
Silica	21.30	26.40	28.95
Alumina	7.65	6.28	11.40
Iron	2.85	1.00	0.54
Lime	60.95	45.22	50.29
Magnesia	2.95	9.00	2.96
Sulphuric acid	1.81	.....	1.37
Alkali	1.15	4.00	.....
Carbonic acid and water	.....	7.86	3.39

As the composition of natural cement from different plants, and frequently from the same plant, varies greatly, the analysis just given must not be considered as an average, but simply as an illustration.

Manufacture.

Raw Materials. The essentials, silica, lime and alumina, are obtained from six different sources.

(1) Cement rock and limestone.

(2) Limestone and clay.

(3) Marl and clay.

(4) Chalk and clay.

(5) Slag and limestone.

(6) Alkali waste and clay.

Cement rock, from which about two-thirds of the cement manufactured in the United States is made, is an argillaceous limestone, low in magnesia.

Marl, an almost pure calcium carbonate, is a soft, wet, calcareous earth.

Clay is a more or less plastic substance composed chiefly of aluminum silicate, formed by the decomposition of minerals.

Chalk, a soft, earthy variety of limestone or carbonate of lime, is usually of a yellowish-white color, but is sometimes snow white. It is easily broken, has an earthy fracture, is rough, dry and harsh to the touch, and adheres slightly to the tongue. It sometimes contains a little silica, alumina, or magnesia, and occasionally all three.

Limestone is a substance formed when clay has been deposited with calcareous matter.

Alkali waste is the refuse from the manufacture of soda. It exists as caustic lime.

These materials are very carefully analyzed and proportioned before mixing. The mixing is done in one of two ways, (1) by a wet or (2) by a dry process, after which the mixture is calcined. For this there are two kinds of kilns in use: (1) the stationary and (2) the rotary. Including the grinding of the clinker, the manufacture of cement, regardless of the process or method used, consists of three steps: (1) mixing and grinding, (2) calcining the mixture, and (3) reducing the clinker to a powder.

References.

"Practical Cement Testing," by Taylor; "Concrete: Plain and Reinforced," by Taylor and Thompson; "Examination of Portland Cement," by R. K. Meade; "Manufacture of Portland Cements," by A. V. Bleininger; "Fourth Series," Bulletin 3, Ohio Geological Survey.

CHAPTER II.

SAMPLING.

Storage. Cement is shipped in wooden barrels, cloth or paper bags, none of which furnish very good protection for its contents. Therefore, it is necessary to provide a good dry storage place.

Inspection. The material, the condition of the packages, and, if possible, the transporting medium of each shipment should be thoroughly examined. Be very careful in the examination of the storage room or warehouse. It must be dry and free from leaks. All packages should bear the manufacturer's name or trade-mark, and any unmarked packages should be rejected. If the specifications call for sealed packages, all packages should be sealed and all seals should be similar. The cement should contain no hard lumps, as these indicate injury from moisture, which has caused partial set. Soft lumps easily broken by the fingers indicate aging, which is not harmful. It is well to ascertain the average weight of the packages.

Collecting the Sample. The selection of the sample for testing must be left largely to the discretion of the party taking it. The quantity must depend upon the importance of the work and the number of tests to be made, as well as the facilities for making them — usually 8 to 10 pounds.

The sample must be a fair average of the shipment and also of the package. One barrel in ten is a fair average for a large shipment, but on small work or in small shipments samples should be taken more often; and never less than five bags should be sampled.

To obtain an average of the shipment, the samples should be taken from packages in different parts of the pile. An average of the package is obtained by means of a sampling auger (Fig. 1), such as is used by butter or sugar inspectors, inserting it from top to center in bags, and from side to center in barrels, midway between the heads.

FIG. 1.

Sample cans * marked with all necessary information as to the shipment, brand, manufacturer, etc., should be used to store the sample in, until tested. Each sample should be thoroughly mixed and passed through a sieve having twenty meshes per linear inch, in order to break up lumps and remove foreign material and further mix the sample.

REFERENCE.

"Practical Cement Testing," by Taylor.

* Mason (fruit) jars make good sample jars.

CHAPTER III.

FINENESS.

Importance. In itself, fineness is of little importance, but because it affects the other properties it becomes of considerable moment.

In the early stages of hardening only the finer particles have any effect, as water is slow in reaching the interior of the larger particles, thereby delaying the hydraulic action. Also, the finer particles will more easily cover the sand grains, making mortar much stronger, and allowing the use

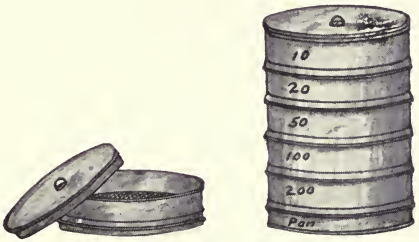

FIG. 2.

of a larger percentage of sand. Neat cement mixtures are usually less strong with fine than with coarse cement. Seasoning can take place more easily with finely ground cement; because of this, fine cement is less liable to unsoundness.

Method. Fineness is determined by passing the cement through sieves (Fig. 2); other methods by means of currents of air or liquids have been proposed but are little used.*

Apparatus. Sieves, numbers 20, 50, 100 and 200, pan and cover, shot and scales. The sieves should be circular, between 6 and 8 inches in diameter and $2\frac{1}{2}$ inches deep, provided with a pan 2 inches deep, and a cover.

The wire cloth should be woven from brass wire having diameters as follows:

	Inches.
No. 20	0.034
No. 50	0.0090
No. 100	0.0045
No. 200	0.0024

The cloth should be mounted on the frames without distortion; the mesh should be regular in spacing and within the following limits:

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.

* "The classification of coarse materials according to size is very readily accomplished by means of sieves." But with cement and other fine materials a large proportion will pass the finest sieve; it is, therefore, desirable to have some way of separating these very fine particles.

"It is well known that homogeneous substances fall in liquids with a speed that varies with their size, that is to say, the larger fall more rapidly than the smaller ones; and if a stream of liquid can be given a definite upward flow, certain relatively coarse particles will settle out and other relatively fine particles will be floated away."

Classifiers for laboratory use have been made for the separation of fine particles, but the experiments so far take from two to four hours, and for cement are otherwise not entirely satisfactory.

See article by G. W. Thompson, Proc. Am. Socy. for Testing Materials, 1910, vol. x, p. 601, also Taylor's "Practical Cement Testing," p. 64.

Scales. The scales used in tests for fineness should be sensitive to 5 centigrams. Figs. 3 and 4 show the two types generally used. That shown in Fig. 4 is designed especially for fineness tests. The beam is graduated so as to represent percentages of 50 grams.*

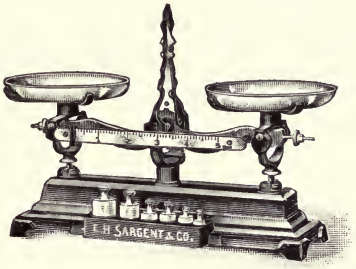

FIG. 3.

To Make the Test. Weigh out 50 grams of the thoroughly dried and coarsely screened sample, and place in the No. 200 sieve with about 200 grams of rather coarse shot. Then,

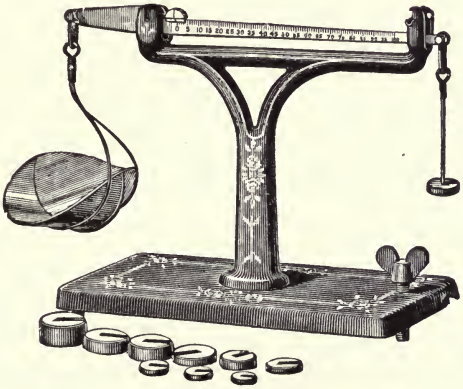

FIG. 4.

with pan and cover attached, hold all in the hands in a slightly inclined position, and move backward and forward,

* The cuts for Figs. 3 and 4 were loaned by the E. H. Sargent Co., of Chicago.

changing the plane of inclination, and allowing the sieve to strike the palm of the hand a sharp blow, squarely on the side, at the rate of about two hundred strokes a minute, for ten minutes. Empty the cement that has passed the sieve, clean the pan, and shake for one minute more.* When the amount passing the sieve in one minute of continuous shaking is less than 0.1 per cent (0.05 gram), the residue is passed through the No. 20 sieve, to separate it from the shot, and is weighed. This weight in grams, multiplied by 2, gives the per cent retained on the No. 200 sieve. Place the residue on the No. 100 sieve and continue the operation as above.

Results should be reported to 0.1 per cent on forms similar to the one shown at the end of the chapter.

A convenient method of determining when less than 0.1 per cent has passed a sieve is to weigh out 0.05 gram of cement; form it into a compact heap and lay it aside for reference. Then, by comparing the cement being tested with this, it can be told at a glance if the shaking should be continued.

The difference in color and structure between the several residues should be observed and noted.

Deductions from Test. Specifications generally state that for natural cement a residue of not more than 15 per cent shall be left on the No. 100 sieve, nor 30 per cent on

* Should the residue after the one minute of continuous shaking be greater than 0.1 per cent the operation must be continued till not more than 0.1 per cent remains on the sieve after one minute of continuous shaking.

the No. 200 sieve; and for Portland cement not more than 8 per cent on the No. 100, nor 25 per cent on the No. 200 sieve; but unless the cement acts badly in the other tests it is not well to reject a cement, except when variation from these requirements is great.

References.

"Taylor's Practical Cement Testing," pp. 75–78; Johnson's "Materials of Construction," Art. 310; "Standard Methods of Testing and Specifications for Cement," Pars. 19–27.

Fineness.

Apparatus.....

Condition of Sample.....

.....

Brand	No.	Weight of Sample	No. 200 Sieve		No. 100 Sieve		No. 50 Sieve	
			Weight Retained	% to Retained	Weight Retained	% to Retained	Weight Retained	% to Retained

Remarks.....

.....

.....

CHAPTER IV.

SPECIFIC GRAVITY.

Definition. The specific gravity of a substance is the ratio of the weight of a given volume of that substance to the weight of an equal volume of water. In the metric system it is the ratio of the weight of the substance in grams to its volume in cubic centimeters.

Significance. "The specific gravity of cement is lowered by adulteration and hydration, but the adulteration must be in considerable quantity to affect the results appreciably."

As the differences in specific gravity are usually very small, every precaution must be taken to make the results accurate. At the best, it is now believed, the test is of but little value, as can be seen from the following, which are the conclusions reached by R. K. Mead and Lester C. Hawk after a series of experiments.*

"The specific gravity test is of no value whatever in detecting underburning, as underburned cement will show a specific gravity much higher than that set by the standard specifications. Underburned cement is readily and promptly detected by the soundness tests, and no others are needed for this purpose.

* Proceedings of the 10th annual meeting of the Am. Socy. for Testing Materials, vol. vii, p. 363.

"The value of the specific gravity test as an indication of adulteration is much exaggerated. While a large admixture of any light adulterant with the cement would be shown, there is at the same time much slag and also Rosendale cement which could be mixed with cement in large quantities without lowering the specific gravity below the limit of our standard specifications.

FIG. 5.

"Low specific gravity is usually caused by seasoning of the cement or clinker, either of which improves the product.

"The proposition to ignite the cement sample which falls below specifications and determine the specific gravity upon the ignited portions is of no value because adulterated cements also have their specific gravity very much raised by such ignition."

FIG. 6.

Apparatus. Le Chatelier specific gravity flask, glass funnel, ring stand, settling jar, glass rod and pipette (Fig. 5), chemical balance (Fig. 20). Various forms of specific gravity flasks have been devised, most of them on the same principle; but the one now used almost exclusively is the Le Chatelier apparatus (Fig. 6).

This consists of a flask (*a*) of 120 c.c. capacity, with a neck (*b*) about 9 mm. in diameter and 20 cm. long, which has a bulb (*c*) at the middle, with graduation marks immediately above and below; the volume between these marks is 20 c.c. The neck, from the mark above the bulb

for about 6 cm., is graduated into tenths of a cubic centimeter.

To Make the Test. Fill the Le Chatelier flask with benzine (62° Baumé naphtha) to the lowest mark, taking care not to wet the side of the neck above the bulb. Let this stand while 65 grams* of the cement to be tested is weighed out on a chemical balance. With a pipette, adjust the lower meniscus exactly to the mark under the bulb taking care to avoid parallax by sighting on a distant horizontal line of about the same level as the eye. Support funnel (*a*) by a ring stand (*b*), (Fig. 7), allowing the stem to project into the flask about one inch. A pad of paper should be placed under the flask for protection from breaking.

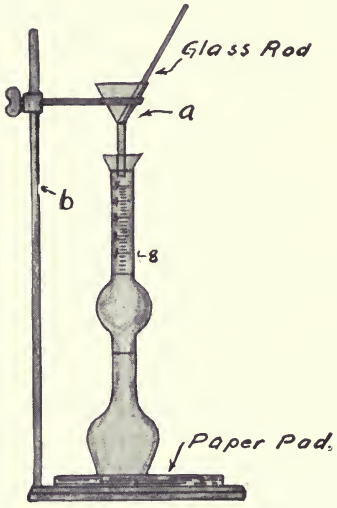

FIG. 7.

Introduce the cement all at one time into the funnel, holding a glass rod in the bottom to control the discharge of the cement. By slightly raising and lowering the glass rod, a small portion of the cement will pass through

* While the Standard Specifications give 64 grams the authors have been in the habit of using 65 grams. Other cases are known in which the test is made with 65 grams of Portland or 64 of natural cements.

the funnel into the flask; and by slightly jarring the flask on the paper pad, all of the cement may be made to pass to the bottom of the flask with almost perfect elimination of air, thereby displacing the benzine. See that all of the cement enters the flask, by brushing scale pan, weighing paper, rod and funnel. The funnel used in introducing the cement must be perfectly dry; otherwise some of the cement will be prevented from entering and the test will be spoiled.

The displaced benzine rises to some division in the graduated neck, as 0.8, shown by the line in Fig. 7; and this plus 20 c.c. (the vol. of bulb), making 20.8 c.c., is the volume of the 65 grams of cement; and

$$\text{Sp. Gr.} = \frac{\text{weight of the cement}}{\text{displaced volume}} = \frac{65 \text{ gms.}}{20.8 \text{ c.c.}} = 3.125.$$

To prevent evaporation the flask should always be grasped above the benzine. The room should be cool and free from air currents. The flask may be immersed in water to keep it at a constant temperature.

Cleaning the Flask. To empty the flask, shake it vigorously to loosen the cement, then invert quickly over a large jar and shake with a vertical motion. Add a small amount of clear benzine and repeat the operation until all the cement is removed.*

The benzine should be filtered and used again.

* A thorough cleaning of the lower bulb is not necessary between tests, but the neck must always be kept clean.

Conclusion. In order to draw conclusions from a specific gravity test, the operator must be familiar with the specific gravity of the brand of cement he is working with, as different brands vary greatly. When a sample tests below the known average of the brand it must be subjected to further examination for adulterants, and an additional specific gravity test should be made on an ignited sample, to see that the low specific gravity is not due to excessive seasoning.

The final rejection or acceptance must be based on the results of the strength tests, modified by the specific gravity, as the experience of the operator indicates.

References.

Taylor's "Practical Cement Testing," pp. 46–51, 58–63; Johnson's, "Materials of Construction," Art. 311; "Standard Methods of Testing and Specifications for Cement," Pars. 8–19.

Specific Gravity.

Apparatus.....

Condition of Sample.....

.....

Brand	No.	Weight of Sample	Increase of Volume	Specific Gravity	Average

Remarks.....

.....

.....

CHAPTER V.

NORMAL CONSISTENCY, MIXING, TIME OF SET.

Significance. Different percentages of water used in making the pastes,* for soundness tests, setting tests, briquettes for strength tests, etc., cause the same sample of cement to give widely varying results. Likewise, these results are affected by a difference in the amount of working that the pat receives. Therefore, it is necessary to fix some standard by which the amount of water may always be kept uniform, and the amount of working always the same.

Standard. The standard requires the use of such a quantity of water as will, with a definite amount of working, reduce the cement to a certain state of plasticity, called normal consistency. The plasticity recommended by the Standard Specifications is such that the plunger of a Vicat apparatus will sink to a point in the mass 10 mm. below the top of the ring.

Apparatus. Vicat apparatus, scales, glass plate and rubber ring, burette (Fig. 8 or Fig. 36). The Vicat apparatus is the one now used almost entirely. This consists of a frame (*k*), (Fig. 9), in which a rod (*l*) moves. There are two caps (*a* and *d*) which may be placed on the upper end of

* The term paste is used to designate a mixture of cement and water, and the term mortar a mixture of cement, sand and water.

the rod (*l*) and a needle (*h*) 1 mm. in diameter, or cylinder (*b*) 1 cm. in diameter (0.39 in.), at the lower end and held by a thumbscrew (*g*). The caps (*d* and *a*) are of such weight that when used with the needle or cylinder respectively, their

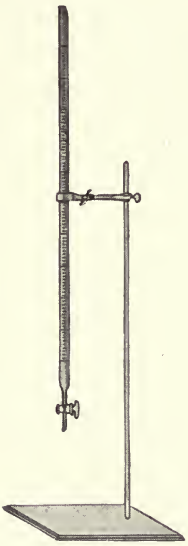

FIG. 8.

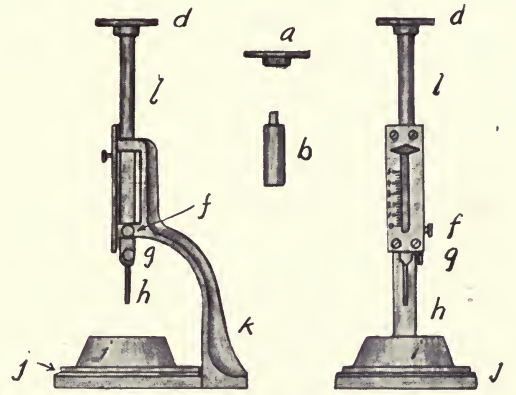

FIG. 9.

weight together with the rod (*l*) is 300 grams. To the rod (*l*), which can be held in any desired position by the thumbscrew (*f*), is attached an indicator, which moves over a scale (graduated to millimeters) attached to the frame (*k*). The paste is placed in a rubber ring (*i*) 4 cm. high and 7 cm. in diameter at the base and slightly tapering to the top, resting on a glass plate (*j*) about 10 cm. square.

Mixing. Weigh out 500 grams of cement and form it into a crater about 4 inches in diameter (Fig. 10). For first trial, into this crater pour, all at one time, a quantity

of water equal in amount to about 20 per cent of the weight of the cement. With a trowel turn the cement from the outside edges, into the water, a little at a time, till the crater is filled and the water is absorbed. This should take about a minute's time. Now turn this mixture over two or three times with the trowel to distribute equally the wet cement, and form into a pile. Knead the mixture vigorously for $1\frac{1}{2}$ minutes, much as dough is kneaded for bread. The process is best described as follows: Place the hands, with the fingers and thumbs touching, over the top of the pile, the wrists resting on the table. Then push rapidly forward with a downward pressure of about 15 pounds and at the same time close the fingers in, so as to squeeze the pile. Do this two or three times. Then turn the pile at an angle of 90 degrees and continue the kneading. At the end of a minute and a half, form the paste into a ball and toss from one hand to the other six times, holding the hands 6 inches apart.*

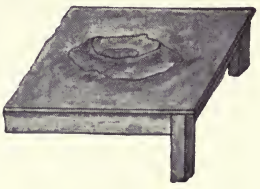

FIG. 10.

To Make the Test. Press the ball into the large end of the rubber ring of the Vicat apparatus, smooth off and place on a glass plate with the large end down. Smooth the top with a trowel, using light pressure. Place the ring under the Vicat plunger; set the plunger in contact with

* To secure uniformity of results, these directions must be followed to the letter.

the surface, read the scale and quickly release the plunger.

Take the final reading on the scale when perceptible motion has ceased. The paste is of normal consistency when the plunger penetrates 10 mm. below the surface. If the correct penetration is not obtained, discard the sample and make another trial, using more or less water as required. Continue until the right percentage of water is found. Record on blanks similar to the one shown at the end of the chapter.

The Vicat apparatus must always be kept exceptionally clean.

During all mixing operations the hands should be protected with rubber gloves.

Time of Set.

Significance. The time of set gives no indication of the strength or soundness of a cement. But it is very important to know the time of initial set (the time which elapses from the moment water is added to the cement until the paste ceases to be plastic, or when crystallization begins), and final set (the time taken to become a hard mass).

Handling the cement after it commences to set, or after the process of crystallization or hardening has begun, will weaken it and cause it to disintegrate. It is, therefore, very important to know how long a time may be allowed in mixing and placing a batch of cement without injury to

it, and after it is placed how long it will take it to harden so that the forms may be removed. It should harden as quickly as possible after initial set. It usually takes considerable time for mixing and placing concrete (the form in which cement is most generally used); therefore, a cement should have a slow "initial set" but reach "final set" soon after.

These periods are arbitrarily measured by the penetration of weighted wires of given diameter, as the needle of the Vicat apparatus.

Things Affecting the Time of Set. Fineness, allowing the water to reach the interior of the particles more easily, increases the rapidity of setting.

Long standing cements absorb moisture from the air and lose their hydraulic property.

The greater the amount of water used in mixing, the slower will be the set.

Increased temperature of the mixing water hastens the time of set.

To Make the Test. The Vicat apparatus with needle is used. Mix 500 grams of cement to a paste of normal consistency. Record the time of adding the water. Place the paste in the Vicat ring as in normal consistency tests, bring the Vicat needle into contact with the surface, and release quickly. Find to what mark the needle should descend to be 5 mm. from the bottom of the ring. Release needle at intervals till set occurs.

Initial set is said to have occurred when the needle

ceases to penetrate beyond a point 5 mm. from the bottom, and final set when it makes no indentation.

Always see that the needle and apparatus are clean.

If Portland and natural cements are to be tested at the same time, mix the Portland first, as it requires more time to attain set. Test Portland cement for initial set after 15 minutes, and natural after 5 minutes.

The rings with the samples being tested should be stored in a damp closet during the test.

Conclusions. Time of set being influenced by so many conditions, tests made by the same operator often do not check closer than 10 per cent and by different operators vary even more. Therefore, considerable allowance should be made in judging a cement for time of set. It requires from 20 to 30 minutes to mix and place a batch of concrete in large work, but it also takes much longer to set than in the tests; so that, in general, a cement need not be rejected unless the mixing and placing on the work takes two or three times as long as the test set.

References.

Normal Consistency. Taylor's, "Practical Cement Testing," pp. 92 to 95, 120 to 126; Johnson's, "Materials of Construction," Arts. 316, 318; "Standard Methods of Testing and Specifications for Cement," Pars. 27, 37, 52, 59.

Time of Set. Taylor's "Practical Cement Testing," pp. 80 to 83, 88 and 89; Johnson's, "Materials of Construction," Arts. 164, 312, 421; "Standard Methods of Testing and Specifications for Cement," Pars. 37 to 44.

Normal Consistency.

Apparatus .....

Temperature of Mixing Water .....

Temperature of Room .....

Humidity .....

Brand	No.	Weight of Sample	Per Cent of Water	Penetration

Remarks .....

.....

.....

TIME OF SET.

Apparatus. .....

Temperature of Room .....

Temperature of Mixing Water .....

Humidity .....

Brand	No.	Water Per Cent	Time of Adding Water	Initial Set		Final Set		
				Clock Time	Elapsed Time	Clock Time	Elapsed Time	

Remarks .....

.....

.....

CHAPTER VI.

CONSTANCY OF VOLUME.

Significance. A cement to be of value must be perfectly sound; that is, it must remain constant in volume and not disintegrate or crumble. Although normal tests give more reliable results, it usually takes considerable time for the natural causes to take effect; and it is necessary to know these effects at once. It has, therefore, become necessary to develop, in some way, those qualities which tend to destroy the strength and durability of a cement.

Tests devised to do this are known as accelerated tests. Failure is revealed by cracking, checking, swelling, or disintegrating, or by a combination of all of these phenomena.

Causes of Unsoundness. Excess of free or loosely combined lime, which has not become sufficiently hydrated, excess of magnesia and alkalies, and sometimes sulphides, insufficient seasoning and coarse grinding are all causes of unsoundness.*

Kinds of Tests. There are two classes of tests, viz., *normal* and *accelerated.* (1) Normal tests are made in either air or water, kept at a constant temperature as near

* For a very complete treatise on soundness, see Taylor's "Practical Cement Testing."

70° as possible. (2) Accelerated tests are made in air, steam or water at a temperature of 115° F. and higher.

Apparatus. Glass plate 4 ins. by 4 ins. by $\frac{1}{8}$ in.; small trowel (Fig. 11), boiling apparatus (Fig. 32).

To Make the Tests. Weigh out 500 grams of cement, and mix into a paste of normal consistency, from which make four pats and one ball.*

FIG. 11.

The pats must be about 3 inches in diameter and from $\frac{3}{8}$ to $\frac{1}{2}$ inch thick at the center, sloping to a very thin, smooth edge at the circumference. In shaping these pats, place on a glass plate about 4 inches square (using no oil on glass) an amount of cement which will almost cover the plate and will measure $\frac{1}{2}$ inch in thickness at the center. Holding the plate in the left hand, shape the cement into a cone with an altitude from $\frac{3}{8}$ inch to $\frac{1}{2}$ inch and a base about 3 inches in diameter. Work the cement to a thin, smooth and uniform surface by troweling and turning the pat, the trowel being inclined from handle to point at an angle (about 18°) which will reduce the thickness of the cement from $\frac{1}{2}$ inch in the center to a very thin edge at the circumference (Fig. 12).

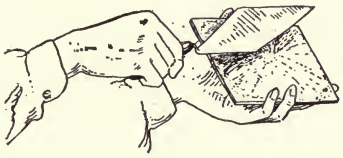

FIG. 12.

To shape the ball, take enough of the paste to form a ball

* The ball test is no longer a standard test.

$1\frac{1}{4}$ inches in diameter and shape by rolling in the hand, as in making a snowball.

Date all specimens, and if different cements are being tested give each pat and ball a distinguishing mark. Store in a damp closet for 24 hours.

For the normal tests, one pat is placed in a water storage tank for 28 days. The water must be maintained as near 70° F. as possible. A second pat must be placed in air maintained at ordinary temperature and humidity. These pats must be observed at intervals of 3, 7, 14, 21 and 28 days, and any changes in their condition should be noted on suitable blanks, as shown at the end of the chapter.

For accelerated tests, place the two remaining pats on the wire screen above the water of the boiling apparatus* (Fig. 32), and the ball on the wire screen in the water. Boil for five hours and examine for signs of failure, such as cracking, discoloration, loosening from the plate, warping, etc., and record condition. The water in the boiling apparatus should be changed often.

Conclusions. To pass satisfactorily, pats should remain hard and firm and show no signs of cracking, distortion or disintegration.

Experience is necessary to judge correctly from the appearance of test specimens whether a cement be rejected or not.† Shrinkage or expansion cracks, small radial cracks at the edge or a short, circumferential split, must

* See description in Chapter X, p. 75.

† Taylor, pp. 178, 179, 182.

not be confused and interpreted as disintegration. These usually come from a too wet mixture or poor working. Leaving the plate cannot be considered as dangerous, unless curling, thickening at the center, or other distortion is very marked. Cracking of the plate, which takes place only in the case of water pats, does not indicate unsoundness. A blotched or discolored pat usually means an adulterated or underburned cement and needs investigation as to the cause.

Usually, if pats, either in the normal or accelerated tests, show only slight signs of failure, it is well to hold the cement for further tests and development of the present one. The further tests should always be made in such a case, as well as a continuation of the first one. Sometimes a new cement which appears unsound in the first test will show perfectly sound after aging a short time.

References.

Taylor's, "Practical Cement Testing," pp. 156, 162, 166, 171; Johnson's, "Materials of Construction," Arts. 313, 314; "Standard Methods of Testing and Specifications of Cement," Pars. 69 to 76.

Constancy of Volume.

Percentage of Water used in Mixing .....

Cement Brand		Age Days	Condition of Pat	Temp.
	Pats in Air	7		
		14		
		21		
		28		
	Pats in water	7		
		14		
		21		
		28		

Condition of Ball .....

Condition of Pat steamed 3 Hours .....

.....

.....

Conclusions .....

.....

.....

CHAPTER VII.

TENSILE STRENGTH.

Use. Cement is used in compression, but since there is a certain fairly definite relation between its strength in compression and tension, the accepted method for determining this strength is the use of a tensile test, which is the test most easily performed. It is made by mixing the cement into a paste, or the cement and sand into a mortar, and molding into test specimens or briquettes, which are allowed to set and are tested at the end of 1, 7 and 28 days, or, in some cases, at longer intervals. Strength tests of mortar briquettes are of much greater importance than are neat cement tests, as it is in the form of a mortar that cement is used.

Factors Affecting Strength.

Composition. Aluminates are presumably responsible for the setting, and silicates for the final hardening; therefore, high aluminates will give a cement a higher early strength and a lower ultimate, and *vice versa*.

Aging. Cement should not age longer than necessary for manufacture, as this will lower the initial strength and, if it is allowed to continue much longer, the ultimate strength.

Fineness. Fineness will, in general, weaken neat cement, but will strengthen sand mortar. This increase in strength of sand mortar is due to the fact that fine cement more thoroughly covers the sand grains, while in the neat the weakening seems to be due to the fact that, in a coarse briquette, the line of break passes around, rather than through the grains, thereby increasing the breaking area.

Amount of Water. The amount of water used in mixing and the method employed seem to affect the strength of the cement greatly. (Chap. V.)

FIG. 13.

Apparatus. Briquette molds, either single (Fig. 13) or gang (Fig. 14), glass plate the size of the mold.

To Make the Test. (Neat Cement.) Weigh out 800 grams of cement (which will make 5 briquettes), mix to normal consistency and fill the molds as

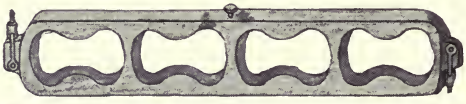

FIG. 14.

follows. (The molding of the briquettes is perhaps the most important factor in cement testing.)

Place a well-oiled mold on an oiled glass plate,* sides toward the operator, put enough cement to half fill the molds into each opening, and press it lightly and evenly

* Plate should be of the same width as the outside of the molds and enough longer to rest on the cleats of the damp closet to be used.

into the bottom of the molds. Do this with the fingers and thumbs, never with a tamper of any sort. Place enough cement in and above the molds to more than fill them; turn the molds 90° and begin at the end farthest away to press, without ramming, the cement into the molds with the thumbs, gripping the sides of the molds with the fingers so as to exert a pressure of about 25 pounds. Press each briquette in this manner three times, once at each end and once in the middle. Turn the mold back to the original position, add more material and smooth off with a trowel, using about 5 pounds pressure. The smoothing should be a cutting action, taking away the excess material and yet filling in all the openings. With a few final strokes of the trowel, make the surface perfectly smooth and press the cement well up to the sides. Place a glass plate on top and turn the mold over, and smooth the bottom in the same manner as the top, by adding material and troweling. The mold resting on the glass plate is now placed in a damp closet for 24 hours, when the briquettes are removed from the molds and stored on edge in water. They must remain here until they are to be broken, which should be done immediately after removal from the water.

Make enough briquettes so that five may be broken at each period called for, usually 1, 7 and 28 days, though often the 1-day tests are omitted.

Mortar Briquettes.

To Make the Test. Weigh out materials as follows:*

For Portland cement, 1 to 3 mortar, cement 250 grams, sand 750 grams.

For natural cement, 1 to 2 mortar, cement 300 grams, sand 600 grams.

Determine the percentage of water by Taylor's formula, as follows:

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

where

X = per cent of water for the sand mixture;

N = per cent of water for the neat cement;

n = parts of sand to one of cement by weight;

S = a constant depending on the character of the sand and consistency desired. (For Ottawa sand, $S = 25$; for bar sand, $S = 27$ to 33; usually use 33.)

The method of mixing and filling the molds is the same as for the neat briquettes, except that the amount of water is determined as above, and the cement and sand are mixed dry to a uniform color before forming into a crater.

Make six briquettes of each cement, three to be tested at each period of 7 and 28 days. Briquettes should be stored in water in a damp closet (the same as the neat), after being marked with the necessary information as to brand, proportions of sand, etc.

* This amount should be sufficient to fill 4 3-gang molds.

Breaking.

Test pieces must be broken as soon as they are taken from the water. Any standard machine (Figs. 21–25) may be used, but it should be supplied with the solid metal clips (Fig. 15), as they are the ones recommended by the Standard Specifications. Clips should be used without cushioning the points of contact. Great care must be observed to center the briquettes in the clips and to see that they are free from sand, in order to avoid cross-strains, which cause clip breaks. Apply the load slowly, as a suddenly applied load may produce vibration or shock, which will cause the briquette to break before the ultimate strength is reached. The Standard Specifications recommend that the rate of application of pressure be 600 pounds per minute. The average value of the briquettes of one sample broken should be taken, with high or low results excluded.

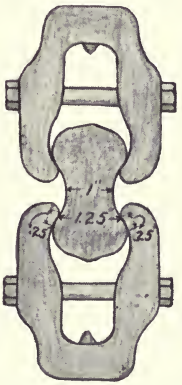

Fig. 15.

Conclusions. A cement, to be acceptable, should fulfil the following conditions in the tension test: " (1) Both neat and sand briquettes shall pass a minimum specified amount at 7 and 28 days. (2) That the neat value at 7 days shall not be excessive. (3) There shall be no falling off between 7 and 28 days in neat test. (4) Sand tests must show an increase of at least 10 to 15 per cent. (5) Sand tests are the true tests of the strength. A cement failing in sand

tests should be rejected even if it passes the neat test. When the reverse is true, i.e., when it passes the sand test and fails in the neat, cement may be accepted if no signs of unsoundness have developed."

The following rules, given by Taylor in "Practical Cement Testing," should be followed.

"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 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."

References.

For Neat. Taylor's "Practical Cement Testing," pp. 120–125; Johnson's, "Materials of Construction," Arts. 315, 316, 319, 320, 323, 324 and 325; "Standard Methods of Testing and Specifications for Cement," Pars. 59–69.

For Mortar. Taylor's "Practical Cement Testing," pp. 108, 114, 120–125; Johnson's "Materials of Construction," Arts. 317–319, 406–411; Taylor and Thompson's "Concrete: Plain and Reinforced," pp. 132, 133; "Standard Methods of Testing and Specifications for Cement," Pars. 35–36.

TENSILE STRENGTH.
(Neat Cement.)

Machine used for Breaking.....

Per cent of Water used in Mixing.....

.....

Age	Brand No.			Brand No.		
	Tensile Strength	Deviation from Mean		Tensile Strength	Deviation from Mean	
		Pounds	Per Cent		Pounds	Per Cent
7 Days						
Mean						

Remarks.....

.....

.....

TENSILE STRENGTH.
(Mortar.)

Machine used for Breaking.....

.....

Brand of Cement	No.	Kind of Sand	Water Per Cent	Proportions	Tensile Strength		Deviation from Mean.			
							Pounds		Per Cent	
					7 Days	28 Days	7 Days	28 Days	7 Days	28 Days
				Mean						
				Mean						

Remarks.....

.....

.....

CHAPTER VIII.

COMPRESSIVE STRENGTH AND TRANSVERSE TESTS.

Compressive Strength. In the United States, compression tests are not used as standard tests for the reception of a cement, but where a concrete is required to be tested, or where a comparison test of different sands and stones that are to be used in concrete is to be made, the compression test is necessary as the size of the aggregate requires the use of larger specimens than the regular briquettes of the tension tests.

The form and size of the specimen most generally used are two-inch cubes for mortar and six-inch cubes for concrete. Cylinders six inches in diameter and ten inches to twelve inches deep are preferable for concrete because the ease of filling and packing them makes the specimens of this size more uniform.

In order that there may be proper contact between the testing machine and the specimen, the bearing surfaces of the specimen should be smoothed to true planes, and to correct any slight angle between the bearing surfaces, one surface should rest on a plate having a ball and socket joint.

Blotting paper or plaster of Paris should be placed between the block and the machine to counteract the irregu-

larities in the specimen; this will, however, slightly lower the strength.

Molds. Four gang 2-inch cube molds (Fig. 16) are generally used, but the size depends to some extent on the capacity of the machines obtainable for breaking the specimens.

To Make the Test. Oil the molds and glass plate thoroughly before mixing.

Neat. Weigh out 900 grams of cement (if 2-inch cubes are to be used) and mix by standard methods, as given

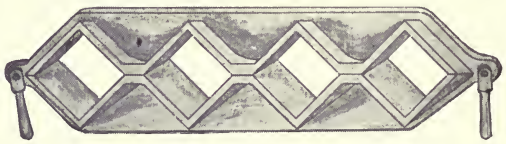

Fig. 16.

under *Normal Consistency.* Fill the molds in a manner similar to that described for briquettes, mark and place in the damp closet for 24 hours. Remove the cubes from the molds and place in water. Do not take from the water until ready to break. Test the cubes at the end of 7 and 28 days.

Mortar. Weigh out 400 grams of cement and 1200 grams of sand for 1 : 3 mortar, or 550 grams of cement and 1100 grams of sand for 1 : 2 mortar.

Determine the amount of water to use and fill the molds, following directions given for mortar briquettes. Place in a damp closet, remove from the molds, store and break, using directions given under *Neat.*

Breaking. Use an ordinary universal testing machine (Fig. 29) of 30,000 capacity (for 2-inch cubes), with compression tool in the moving head. Place the cube exactly in the center of the machine. (This can be done by means of the circles cut in the table of the machine.) Run the head down until nearly in contact with the cube, with medium speed; change to the slowest speed and apply load, keeping the beam balanced until the cube yields under the load.

Conclusion. The results of compressive tests are to be interpreted in accordance with the same general rules as for tension tests. There is a ratio between compression and tensile strength, which varies from 5 to 10 for average results.*

Transverse Tests.
(*Modulus of Rupture.*)

Molds. The molds for this test are usually 1-in. by 1-in. by 13-in. beam molds, though sometimes $1\frac{1}{2}$-in. by $1\frac{1}{2}$-in. by 13-in. or 2-in. by 2-in. by 13-in. molds are used; 6 beams will give a very good average result. Fig. 17 shows a gang mold.

To Make the Tests. Oil the molds and the glass plate† thoroughly. Weigh out 1000 grams of cement; mix and fill the molds in the same manner as for briquettes or cubes. When using gang molds do not try to turn the molds over,

* See discussion in "Materials of Construction" by J. B. Johnson, and Practical Cement Testing, p. 215.

† If glass plate large enough for gang mold is wanting, a water-soaked asbestos board may be used.

but use extra precautions to see that the cement fills the bottom of the molds. Place in damp closet for 24 hours; remove from the molds and place in storage water for 7 days, and then break.

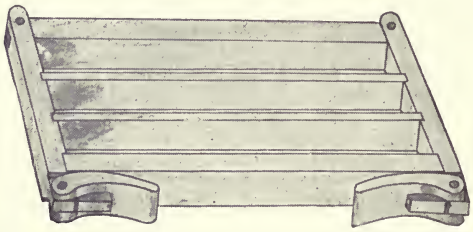

FIG. 17.

Breaking. Beams should be broken on the long lever testing machine with special attachment (see description on p. 70), using, if possible, a 12-inch span. Should any beams be broken in handling before being tested, they may be broken at shorter spans. Reports of this test should include the calculations.

Calculations. The modulus of rupture is calculated by the formula

$R = \frac{3 \cdot W.L}{2 \cdot B.H^2}$, which for 1-inch square specimens becomes

$R = \frac{3}{2} W.L$, in which

W = the center load in pounds,
L = the length of span in inches,
B = width of the specimen,
H = the depth.

Illustration. Suppose a beam 1 in. by 1 in. on a 12-inch span breaks with a center load of 50 pounds; then

$$R = \frac{3}{2} W.L = \frac{3}{2} \times 50 \times 12 = 900 \text{ inch-pounds.}$$

Another beam 2 ins. by 2 ins. on a 12-inch span breaks with 400 pounds, center load.

$$R = \frac{3 \cdot W.L}{2 \cdot B.H^2} = \frac{3 \times 400 \times 12}{2 \times 2 \times 4} = 900 \text{ inch-pounds.}$$

Conclusion. There is a ratio* between transverse strength and tensile strength varying between 1.3 and 2.5. By assuming an average of 1.5 and multiplying the modulus of rupture by this average, an approximate estimate of the tensile strength may be obtained. However, this is rather unsatisfactory, since transverse specimens are subject to so many variations. A cement should never be condemned by a transverse test alone, unless a series of tests shows a very low value.

References.

Compression Tests (*Neat*). Taylor's "Practical Cement Testing," pp. 212–216; Johnson's "Materials of Construction," Arts. 280, 315, 326. (*Mortar*) Taylor, pp. 212–216; Taylor and Thompson's "Concrete: Plain and Reinforced," p. 136; Sabin's "Cement and Concrete," Art. 52.

Modulus of Rupture. Taylor's "Practical Cement Testing," pp. 216, 217, 231–234.

* The student should compare tension and transverse tests and ascertain the ratio.

COMPRESSIVE STRENGTH.
(Neat Cement.)

Machine used for Crushing.....

.....

Per Cent of Water used in Mixing.....

Brand	No.	Age Size	Ultimate Load	Compressive Strength	Deviation from Mean Strength	
					Pounds	o/o
		7 Days				
		Mean				
		28 Days				
		Mean				

Remarks.....

.....

.....

COMPRESSIVE STRENGTH.
(Mortar.)

Machine used in Crushing.....

.....

Cement Brand	No.	Sand Kind	Water o/o	Proportions	Age Days	Ultimate Load	Compressive Strength	Deviation from Mean Strength	
								lbs.	o/o
					7				
					Mean				
					28				
					Mean				

Remarks.....

.....

.....

MODULUS OF RUPTURE.
(Neat.)

Brand of Cement.....

Per Cent of Water used in Mixing.....

Machine used in Breaking.....

.....

Section of Beam	Span	Central Load	Modulus of Rupture lbs. per sq. inch.
Mean			

Remarks.....

.....

.....

CHAPTER IX.

SAND AND STONE.

Sand.

The variation in the strength of mortars, due to different kinds of sand, is so great that to obtain uniformity of tests in different laboratories and by different workers in cement, it was necessary to adopt a standard sand. There are two in use: one, a standard quartz, open to objection on account of its high per cent of voids, the difficulty of compacting in the molds, and lack of uniformity; the other, a natural sand from Ottawa, Illinois, screened to pass a sieve 20 meshes per linear inch, and retained on a sieve having 30 meshes per linear inch. The Ottawa sand is recommended by the committee on Standard Specifications, and should be the one used.

Test of Natural Sand. When a natural sand is to be used for tests or work, it becomes necessary to determine the percentage of voids, the uniformity coefficient and the effective size, in order to properly proportion the sand and cement. It is also well to know the amount of foreign matter, as loam, clay and organic substances, to determine its effect on the strength.

Per Cent of Loam. (Apparatus: Scales, settling jar, glass stirring rod, evaporating dish, ring stand or tripod,

Bunsen burner). Weigh the sand very carefully and place it in a settling jar. Add about 250 c.c. of water and stir vigorously with a glass rod, allow fifteen seconds for settling and decant the water, taking care that none of the sediment at the bottom escapes. Repeat the operation until the water remains clear after fifteen seconds of stirring; wash the entire contents of the jar into a shallow pan or evaporating dish; drain off the water, place the pan and contents on a ring stand over a Bunsen burner (Fig. 18), and thoroughly dry. When cool, weigh and determine the per cent of loam. The loss in weight is the weight of the loam.

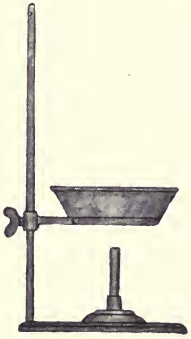

Fig. 18.

Mechanical Analysis.

The mechanical analysis of a sand or a stone consists in determining the fineness (by passing through various sieves), the uniformity coefficient and the effective size.

Apparatus. Sieves 7 inches in diameter, numbers 200, 150, 100, 70, 60, 50, 40, 30, 20, 10, with pan and cover; scales and mechanical shaker. (See p. 74.)

To Make the Test. Weigh out 100 grams of sand, arrange nest of sieves with largest on top, if a mechanical shaker is to be used; if the shaking is to be done by hand, only two or three sieves can be used at a time.

Put the 100 grams of sand in the top sieve, cover and

tighten the sieves in the machine and run it for 5 minutes. A like time of shaking is required for each set of sieves taken

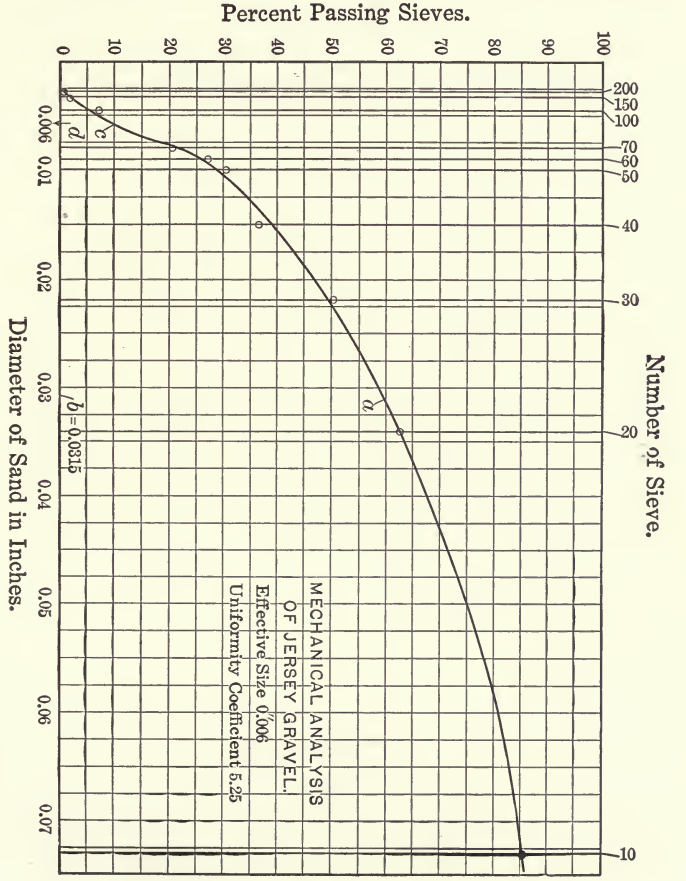

FIG. 19.

at a time, when shaken by hand. Weigh the amounts retained on each sieve and caught in the pan. Record in blanks similar to the one shown, and plot curve (Fig. 19),

showing size of sieves as abscissas and the per cent passing sieves as ordinates. From the curves, determine the effective size and uniformity coefficient.

The uniformity coefficient is the ratio of the diameter of the particles represented at the point where the curve crosses the 60 per cent line, to the diameter of the particles represented at the point where the curve crosses the 10 per cent line.*

The effective size is the size of the particles at the 10 per cent point on the curve.

To Find the Uniformity Coefficient. Project down from the point *a* on the curve (Fig. 19), where the 60 per cent line intersects the curve, to the point *b* on the scale of sand diameters, and likewise from point *c* to *d*. Divide the reading at *b* by that at *d* and the result is the uniformity coefficient, or 0.032 ÷ 0.006 = 5.25.

The effective size is the reading at the point *d*.†

Voids are the spaces between the grains of sand or pieces of crushed stone.

Apparatus. Le Chatelier flask, funnel, glass rod, pipette, wooden rammer and small settling jar.

To Make the Test. Weigh out 55 grams of the sand and determine its specific gravity as directed in Chapter IV, substituting water for the benzine in the Le Chatelier flask.

Determine the weight of sand per unit of volume (1 c.c.)

* Sand having a coefficient over 4.5 is a good coarse sand. The larger the value, the better the sand.

† The effective size is but little used in concrete.

by filling a jar of known capacity* (about 1 liter, 1000 c.c.) with thoroughly dry sand. Fill the jar by introducing a layer of sand 1 inch thick and compacting it with a wooden rammer, add another layer and tamp, and so on, till the jar is level full. Get the net weight of the sand; i.e., weigh the jar and sand, subtract the weight of the jar, and compute the weight of 1 c.c. The weight of sand in grams divided by the volume in cubic centimeters will give the weight of sand per cubic centimeter.

Example:

Weight of jar and sand	2650 gms.
Weight of jar	980 gms.
Weight of sand (1000 c.c.)	1670 gms.

Weight of 1 c.c. = 1.67 gms.

With the weight of 1 c.c. and the specific gravity, compute the per cent of voids as follows: Divide the weight of sand per cubic centimeter by the specific gravity and multiply by 100. This product subtracted from 100 gives the per cent of voids, or

$$100 - \frac{\text{weight of sand per c.c.}}{\text{specific gravity}} \times 100 = \text{per cent of voids}$$

Example:

Weight of sand per c.c. = 1.67
Specific gravity = 2.7

$$100 - \frac{1.67 \times 100}{2.7} = 38 = \text{per cent of voids.}$$

* The capacity can be obtained by filling the jar with a measured amount of water or by filling with water and getting the weight of the water in grams, when each gram will equal a cubic centimeter of volume.

Weight per Cubic Foot. With the per cent of voids just obtained and the specific gravity, the weight per cubic foot can be computed, as follows: Multiply the weight of a cubic foot of water (62.35 lbs.) by the specific gravity, and this product by 100 minus the per cent of voids. The result is the weight per cubic foot, or

62.35 lbs. (wt. of cu. ft. of water) × sp. gr.
× (100 − per cent of voids) = wt. per cu. ft.

Example:

62.35 lbs. × 2.70 × (100 − 38) = 104.16 lbs.

Stone.

Mechanical Analysis.

Apparatus. Sieves with pan and cover (same as those used for sand); also, 7-inch sieves with 0.15-inch, 0.20-inch and 0.30-inch openings, and 12-inch sieves with 0.45-inch, 0.67-inch and 1.00-inch openings. A mechanical sifter will greatly lessen the work of the test.

To Make the Test. Weigh out 10 pounds of crushed stone, and place it on the 1.00-inch sieve; shake over a sheet of paper till none will pass; weigh that retained on the sieve; put that caught by the paper in the 0.67-inch sieve, shake and weigh as before; repeat with the 0.45-inch sieve. Arrange the sieves used in the sand tests, together with the 0.30-inch, 0.20-inch and 0.15-inch in order of their size, the largest on top, in the mechanical shaker, and place the shakings from the 0.45-inch sieve in the top one. Shake for five minutes and find the weight

retained on each sieve and caught in the pan. Plot curve for the stone, using the same method as for sand, and determine the uniformity coefficient and efficient size.

Voids.

First Method.

Apparatus. Chemical balance with specific gravity bench, small beaker, cubic foot measure and platform scales.

Specific Gravity. Determine the specific gravity by the loss of weight in water method; that is, weigh on the chemi-

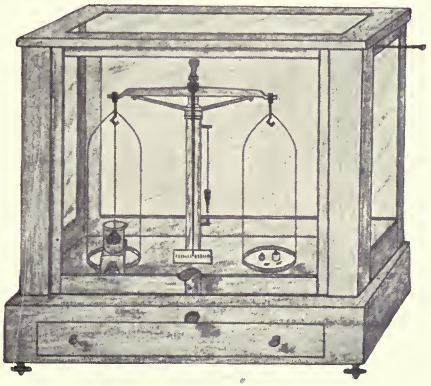

Fig. 20.

cal balance (Fig. 20), a piece of the stone, first in air, and then suspended in water. The specific gravity will be the result obtained by dividing the weight in air by the loss of weight in water, or

$$\text{Sp. gr.} = \frac{\text{weight in air}}{\text{loss of weight in water}}.$$

To Make the Test. Determine the weight in air in the ordinary manner. To get the weight in water, tie a very fine thread around the stone, arrange the balance bench and beaker as shown in Fig. 20, see that the stirrup and pan are free from the bench, and have the beaker about three-quarters full of water. Tie the free end of the thread to the stirrup hook — so that when balanced the stone will hang about the middle of the water, and will not touch the sides of the beaker — and weigh. Subtract this weight from the first, which will give the loss of weight in water. Divide the first weight by this loss of weight in water and the result will be the specific gravity.

Example:

Weight in air	25.02 gms.
Weight in water	15.76 gms.
Loss of weight in water	9.26 gms.

$$\text{Sp. gr.} = \frac{25.02}{9.26} = 2.7$$

Weight of the Stone per Cubic Foot. Use the cubic foot measure. First get its weight empty. Then fill it with the stone, add a layer of about 3 or 4 inches at a time, tamp each layer well with a light wooden rammer and weigh. Subtract weight of the measure and get the net weight of the stone.

Example:

Weight of measure and stone	104 lbs.
Weight of measure	6 lbs.
Net weight of stone	98 lbs.

With this net weight and the specific gravity found above, calculate the per cent of voids. The per cent of

solid content is the weight of stone in pounds (1 cu. ft.) in the measure, multiplied by 100 and divided by the product of the weight of a cubic foot of water (62.35 lbs.) and the specific gravity of the stone. This subtracted from 100 gives the per cent of voids, or

$$100 - \frac{\text{wt. of stone (in measure) in lbs.}}{\text{sp. gr. (of stone)} \times 62.35 \text{ (wt. of cu. ft. of water)}} \times 100 = \text{per cent voids.}$$

Example:

Weight of cu. ft. of stone ..... 98 lbs.
Sp. gr. of stone 2.7

$$100 - \frac{98 \times 100}{62.35 \times 2.7} = 42 = \text{per cent of voids.}$$

Second Method.

This method is more adapted to use in the field and is probably more accurate than the method just described.

Apparatus. Platform scales, gallon jar or 8- to 10-quart pail.

To Make the Test. Weigh the pail (which must be perfectly dry and clean), fill the pail brim full of water and weigh again. Get the net weight of the water and calculate the volume of the pail. (Weight of water in pounds $\times$ 0.01602 = volume in cubic feet). Empty out the water, dry the pail and fill it level full with the stone to be tested, having it well tamped. Get the net weight of the stone. Without changing the stone in any manner, pour into the pail of stone as much water as it will hold (pour slowly to allow the air to escape) and get weight of the

water added and its volume. The percentage of this last volume of water to the first will be the percentage of voids in the stone. Calculate the weight of a cubic foot of the stone from the weight and volume found in the first weighings. The specific gravity can be found by substituting these values in the following equation:

$$\frac{\text{wt. of cu. ft. of stone (solid)}}{\text{wt. of cu. ft. of water}}$$

or

$$\text{wt. of cu. ft. (crushed stone in lbs.)} \times \frac{100}{100 - \text{per cent voids (solid content)}} \div 62.35 \text{ lbs. (wt. of cu. ft. of water)} = \text{specific gravity.}$$

Example:

	Pounds.
Weight of pail and water	34.50
Weight of pail	4.5
Weight of water	30.00
Weight of pail and stone	47.5
Weight of pail	4.5
Weight of stone	43.00
Weight of pail, stone and water	61.00
Weight of pail and stone	47.50
Weight of water in the voids	13.50

Volume of the pail = $30 \times 0.01602 = 0.4806$ cu. ft.
Volume of the voids = $13.5 \times 0.01602 = 0.216$ cu. ft.
Per cent of voids = $0.216 \times 100 \div 0.481 = \mathbf{44.9}$.
Weight of a cubic foot of the crushed stone = 43 lbs. $\div$ 0.481 cu. ft. = **89.4 lbs. per cu. ft.**
Specific gravity is

$$\frac{89.4 \times 100}{62.35 \times (100 - 44.9)} = \frac{8940}{3435.485} = \mathbf{2.6.}$$

References.

Per cent of Loam, Taylor's "Practical Cement Testing," p. 223; Baker's "Masonry Construction," Art. 114 *c*; Taylor and Thompson's "Cement: Plain and Reinforced," p. 154; Mechanical Analysis, Taylor's "Practical Cement Testing," p. 223; Taylor and Thompson's "Concrete: Plain and Reinforced," pp. 187–194; Turneaure and Russell's "Public Water Supply," Art. 511; Voids in Sand and Stone, Taylor's "Practical Cement Testing," pp. 224, 225; Baker's "Masonry Construction," Arts. 114 *f*, 115 *d*; Taylor and Thompson's "Concrete: Plain and Reinforced," p. 210.

Percentage of Loam.
(Sand.)

Kind of Sand	Weight of Sample	Weight of Dry Resid.	Weight of Loam	Per Cent of Loam
Mean.				

Remarks .....

.....

.....

Example:

Weight of sand before washing	50 gms.
Weight of sand after washing	49 gms.
Loss	1 gm.

Hence per cent of loam = 2.

MECHANICAL ANALYSIS.

Sieves No.	Diam of Openings in Inches	Sand:		Stone:	
		Weight Retained	Per Cent Retained	Weight Retained	Per Cent Retained
	Passed #200				
	Total				
	Loss				

SAND:
Effective Size.....
Uniformity Coefficient.....

STONE:
Effective Size.....
Uniformity Coefficient.....

Weight of Sand per Cubic Foot.

Capacity of Jar	Wt. of			Condition of Sand	Wt. per Cu. Cm.
	Jar	Jar + Sand	Sand		

Specific Gravity of Sand.

Kind of Sand	Wt. of Sample	Displacement.	Specific Gravity	Av. Specific Gravity

Specific Gravity of Stone.

Weight of Stone		Displacement.	Specific Gravity	Av. Specific Gravity
In Air	In Water			

WEIGHT OF CRUSHED STONE PER CUBIC FOOT.

Wt. of			Condition of Stone	Wt. per Cu. ft.
Measure	Measure + Stone	Stone		

CHAPTER X.

LABORATORY EQUIPMENT FOR PHYSICAL TESTS.

THE special pieces of apparatus for the different tests have been described under the various tests in which they were used. In this chapter, brief mention will be made of the laboratory equipment now in general use.

MACHINES AND ACCESSORIES.

Tension Tests. There are three types of machines used in the United States for briquette breaking (tension tests); namely, long lever, shot and spring balance.

The long lever machine shown by Figs. 21 and 22 is the most accurate, and has the advantage of being adapted to transverse and small cube tests.

The Olsen (Fig. 21) and Riehle (Fig. 22) are the two makes generally used. The poise on these machines is moved out, either by power or by hand, and can be regulated to travel at different rates of speed. The load is applied by turning a large hand wheel in the center of the machine, which operates a lever, — thereby putting tension on the briquette.

The shot machines (Fig. 23–25) are compact and speedy, and operate without constant attention. The Fairbanks

(Fig. 23), Riehle (Fig. 24) and Olsen (Falkenau-Sinclair) (Fig. 25) are the representative machines of this type.

A description of the operation of the Fairbanks machine

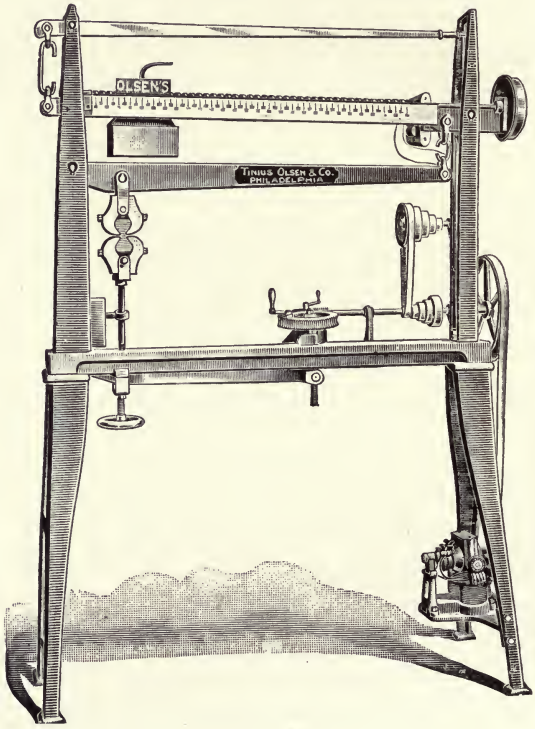

FIG. 21

is as follows: Place a briquette in the clips (*N*)(*N*) (Fig. 23) and tighten up the hand wheel (*P*) till the beam is raised to (*K*), open the shot outlet (*I*); the weight of the shot in the pail (*F*) will put tension on the briquette through the

lever system, till it breaks. The flow of the shot is automatically stopped by the dropping of the beam when the

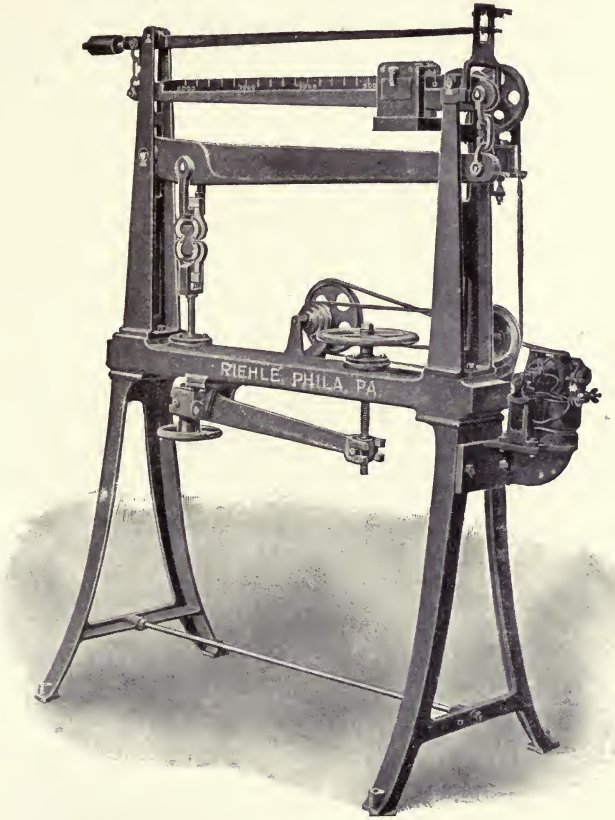

FIG. 22.

briquette breaks. The pail and shot are now hung on (*E*), poise (*G*) is placed on the beam where the pail was, and the breaking load is read as in an ordinary weighing operation.

The Riehle (Fig. 24) is slightly different. A weight on one end of a lever is counterbalanced by shot; as this shot is allowed to pass out, the weight is allowed to descend, thereby putting tension on the specimen. When the specimen breaks, the flow of shot is automatically stopped. The shot is weighed on a spring balance so calibrated as to

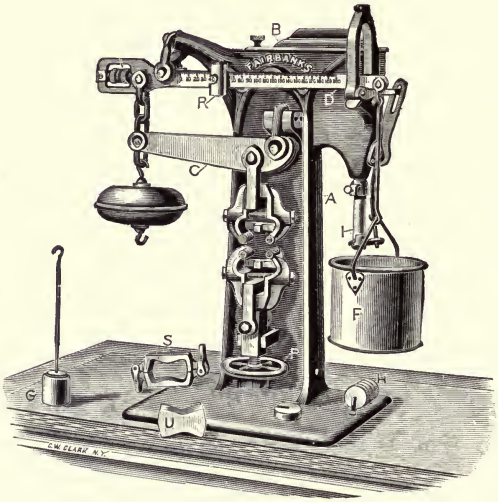

FIG. 23.

read the breaking load directly. These machines can only be used in tension.

The operation of the Falkenau-Sinclair or Olsen shot machine (Fig. 25) is as follows, as described in the catalog of Tinius Olsen & Co.: " Referring to the cut, it will be seen that the machine consists, as usual, of a mechanism for applying the load on the test briquette, and means for weighing

this load. The load is applied through a system of levers by means of the weight shown on the extreme right. Before

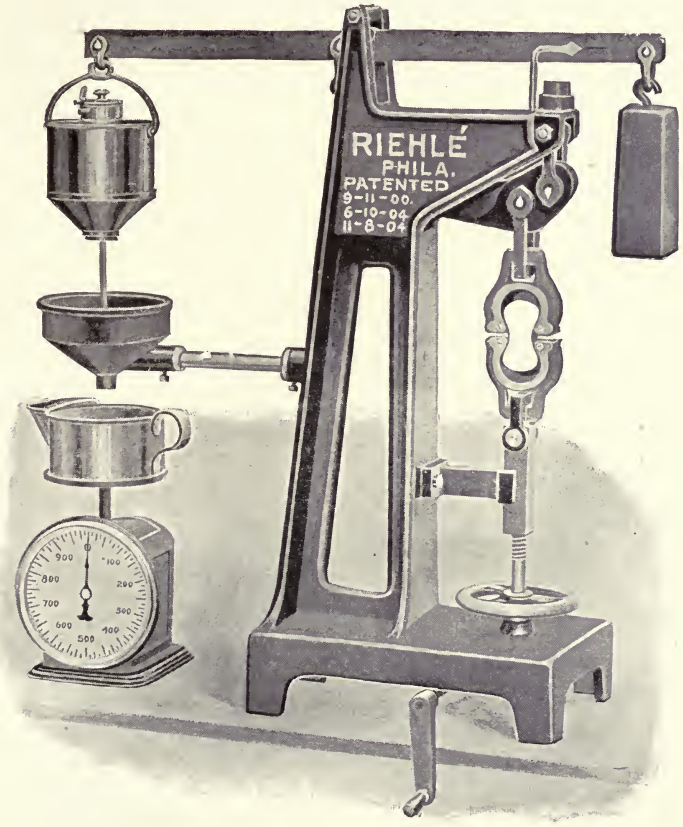

FIG. 24.

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. At the instant the briquette breaks the escaping stream of

Fig. 25.

shot is cut off by the closing of the valve in the bottom of the kettle. This is effected 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 briquette having broken, and the stream of shot having been cut off, it only remains to weigh the amount of shot that has escaped from the pan and multiply it by the proper factor to give the load per square inch to which the briquette has been subjected.

"This shot might be weighed on any scale desired, but for facility in making tests, the machine is furnished with a spring balance on which is placed the pan into which the shot falls. The dial of this spring balance is graduated, so as to read in terms of pounds to the square inch on the specimen. It follows that, as the test proceeds, the operator can watch the application of the load, and knows at any instant exactly what the load is on the briquette. When the briquette breaks, the load which broke it is read at a glance and jotted down without further manipulation or calculation. The shot is then poured back into the kettle and the machine is ready for the next test.

"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. This hand wheel having been properly adjusted, there can be no strain on the briquette by pulling too hard on the hand wheel."

The crank at the base is for taking up the motion of the lever system as the strain increases.

The spring balance machines (Fig. 26) are compact and

cheap, but are neither accurate nor speedy, and are suitable only for field tests or where only occasional tests are made.

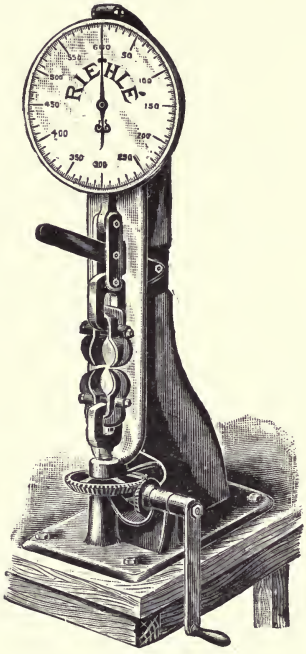

FIG. 26.

Clips. There are several forms of clips on the market, but the one recommended by the committee on standard specifications is to be desired. Its construction may be seen from Fig. 15.

Transverse test attachment (Fig. 27) consists of a bar (*a*) about 13 inches long, with link (*b*) attached at the center so that it can be suspended from the beam of the machine in place of the upper tension clip. V's are cut in bar (*a*) 2, 3, 4, 5 and 6 inches distant from the center on each side, and in these the stirrups (*c*) are placed, according to the span of the test specimen. The test specimen (*d*) is placed on stirrups (*c*–*c*) and a third stirrup (*e*), which is attached to the hand wheel lever in place of the lower tension clip applies the load at the center of the specimen. (Points *f* are blunt knife edges).

Compression Tool. This attachment consists of four plates about $3\frac{3}{4}$ inches square. The plates (*a*) and (*d*) (Fig.

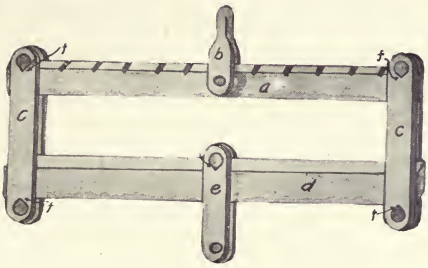

FIG. 27.

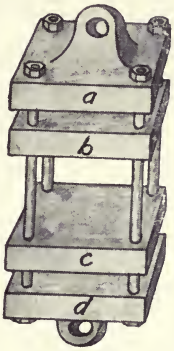

FIG. 28.

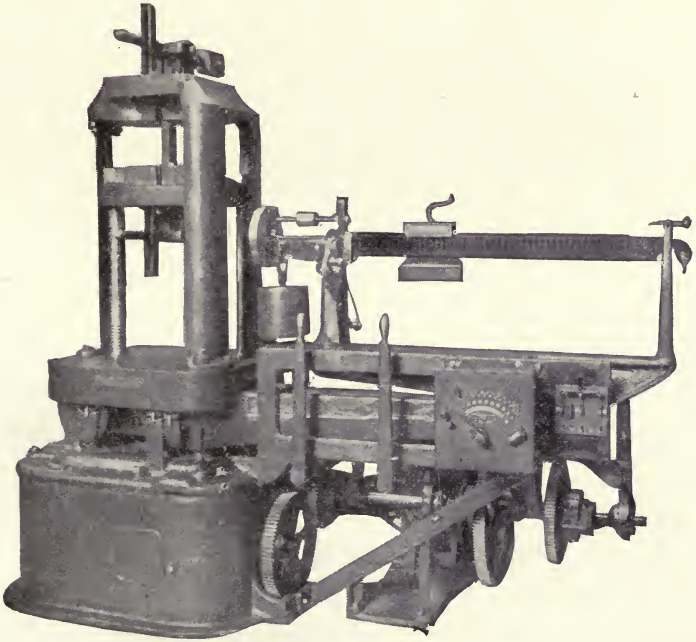

FIG. 29.

28) have ears in the center, by which they are attached to the machine, in place of the tension clips. Plate (*c*) is connected to (*a*), and (*b*) to (*d*), by four bolts, in such a way that when (*a*) and (*d*) move apart, (*b*) and (*c*) will move towards each other and compress the cube between them.

Universal Testing Machine. The universal machine (Fig. 29) is better adapted for compression tests than the long lever machines and has the further advantage of very wide range in the size of blocks.

Figure 30 shows the Olsen Hydraulic Compression Machine. This machine is a small hydraulic press, having an adjustable head above, which can be lowered or raised to the desired position by means of a hand wheel and screw. The cylinder is placed below and pressure is applied to the ram in a peculiar manner. Instead of the usual pump, a small plunger is forced into the cylinder by means of a worm drive shown at the right-hand side. The motion of the ram is limited, as the plunger is of small diameter, but in testing incompressible material, like cement, a short stroke is all that is required. The worm can be slipped out of mesh with the worm wheel, and the plunger can be quickly moved in either direction by means of the hand wheel. The table is on a spherical bearing, which accommodates itself to the specimen so as to bring a uniform pressure. The load is read directly by a hydraulic gage connected with the cylinder through a safety valve, which prevents injury to the gage by sudden shock due to failure. The capacity of the machine is 200,000 pounds. The diam-

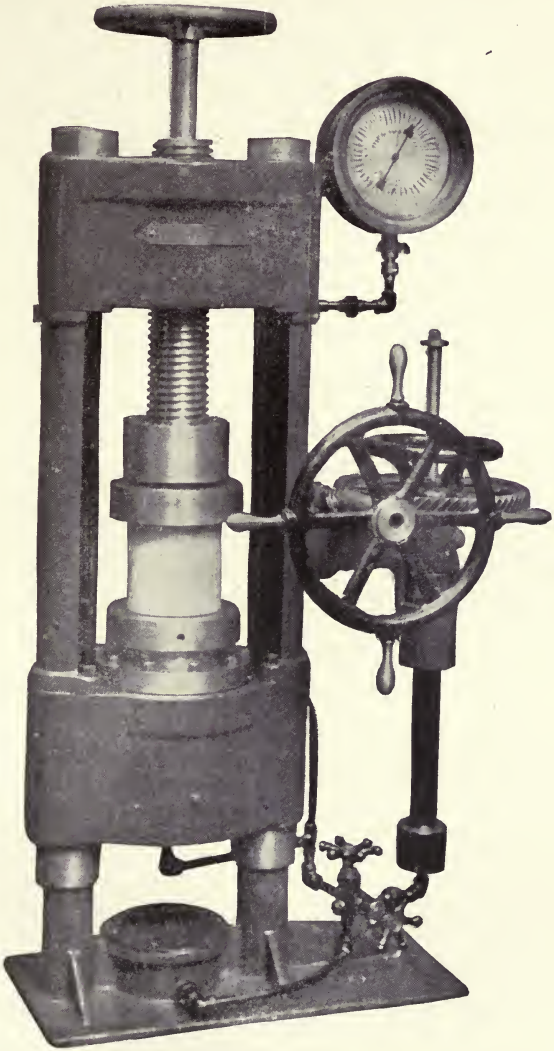

FIG. 30.

eter of the head and table is 6 inches, while the extreme distance between the head and table is $6\frac{1}{2}$ inches.

Sand Sifter. The sifter shown in Fig. 31 is a very close attempt at producing the desired motion for sifting by giving the sieves a circular motion and also a jar. It

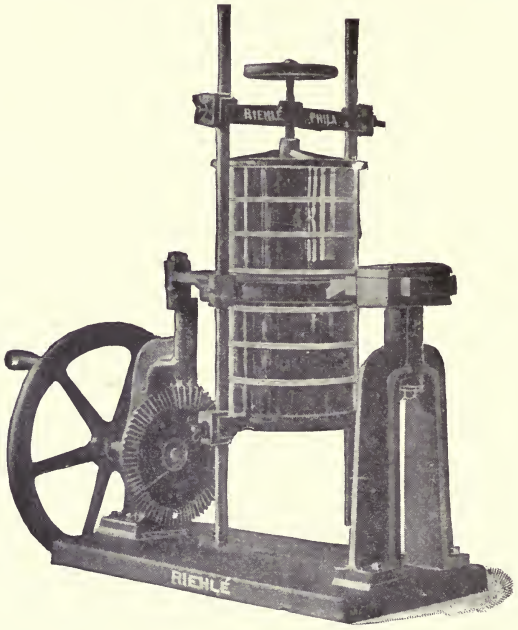

Fig. 31.

consists of bevel gears driven by a hand wheel, which imparts a circular and an up and down motion to two upright rods, between which the sieves are securely held. These rods extend downward so that on the down motion they strike the bottom plate of the machine a sharp blow.

A very good homemade mechanical sifter can be made when occasion demands. A simple one is shown in "Practical Cement Testing," page 73.

Scales and Balance. The laboratory must have a chemical balance (Fig. 20). It need not be an expensive make. A small balance sensible to 5 centigrams (Fig. 3) is used for the general weighing, and ordinary platform scales will be needed for test of concrete.

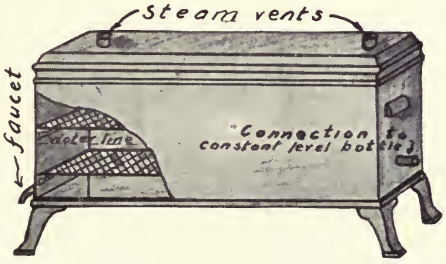

FIG. 32.

Boiling Test Apparatus. This apparatus can be as simple or as elaborate as desired. It need consist only of a copper vessel, with a cover in which there is a small outlet for excess steam and two wire netting shelves, one near the bottom, the other above the water line. It is an advantage to have an opening near the bottom, for a connection to a constant level bottle. The vessel should have legs, or be

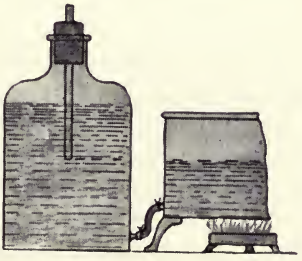

FIG. 33.

placed on a tripod, so that a Bunsen burner can be placed underneath for heating. Fig. 32 shows a very good design. Fig. 33 shows a constant level bottle.

Moist Closet. The moist closet (Fig. 34) is best made of cement, slate or similar material, and may be any size desired. It should be long enough to take in the longest size of gang molds used. The ends should have cleats so arranged that glass strips with molds on them can be slipped in, with enough room between tiers for free circulation of

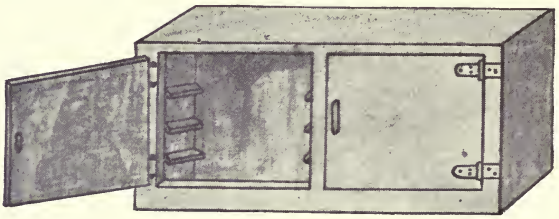

FIG. 34.

air. A pan for holding water should be placed in the bottom. A most excellent closet made of waste cement is described by E. B. McCready, vol. vii, p. 598, Proc. of Am. Socy. for Testing Materials. Ordinary tin boxes are sometimes used for damp closets. A temporary damp closet can be made by bending a wire screen to form a roof over the specimens, over which a wet cloth is placed. There should be some means of keeping the cloth uniformly damp, as letting the ends dip into a pan of water.

Storage Tanks. Any tank that will hold water and has capacity enough can be used for a storage tank. Fig. 35

shows the very elaborate tanks in the Lesley Cement Laboratory at the University of Pennsylvania.

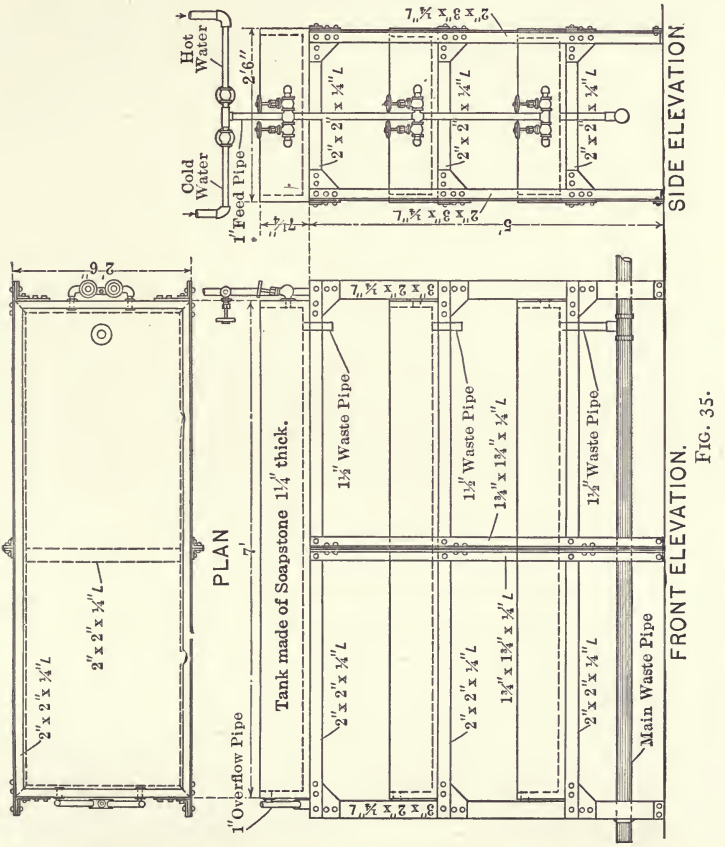

FIG. 35.

Table. Any ordinary strong table may be used, but it must have a mixing plate of slate, marble or glass.* A

* The writer believes the ordinary laboratory bench is too high for good and comfortable working, and therefore advises the use of a bench or table about 3 ft. 1 in. to 3 ft. 3 ins. high.

special table, 2 feet 6 inches wide and about 3 feet 3 inches high, covered with a glass plate and having a tin spout leading from the top, at the back, or one end to a refuse can is very convenient. The spout must be open and 5 or 6 inches wide; otherwise it is likely to clog.

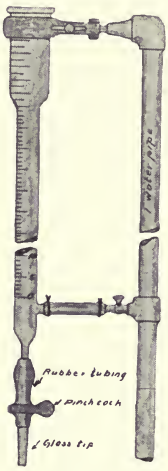

FIG. 36.

Burette (Fig. 8). This is one of the indispensable pieces of apparatus in a cement laboratory. It is used almost constantly for measuring the water used in mixing and for other purposes. Fig. 36 shows the burette used in the Lesley Cement Laboratory, University of Pennsylvania. A water pipe is connected to each bench to which the burette is attached.

Briquette molds should be made of brass, bronze, or some equally noncorrodible material, and have sufficient metal in the sides to prevent spreading during molding. There are two types of molds, single (Fig. 13) and gang (Fig. 14). Gang molds, permitting the molding of 3, 4 or 5 briquettes at a time, are to be preferred, since a greater quantity of mortar can be mixed at one time, giving more uniform results.

Cube molds are shown in Fig. 16. What has been said in regard to briquette molds applies equally well to cube molds.

Bar molds, shown in Fig. 17, are used for making bars for the transverse test. Like the other molds, they must be strong and noncorrodible.

Miscellaneous articles, such as are found in all physical and chemical laboratories, are needed in the testing of cement. Most of these, if not all, have been mentioned under the tests in which they are used, but they are repeated in the following list: glass rod, glass tubing, small-stemmed glass funnel, $\frac{1}{2}$-inch camel's hair brush, pipette, scoop, thermometer, ringstand, tripod, Bunsen burner, evaporating dish, oil can and light machine oil, settling jar, bottles of various sizes, trowel, etc.

For list of equipment needed for a cement laboratory, or for a simple field outfit for a contractor, see "Practical Cement Testing," pp. 239–240.

PART II.

THE CHEMICAL ANALYSIS OF CEMENT, LIMESTONE, MARL, ETC.

It is doubtful if chemical analysis plays any part in the testing of cement for defects in manufacture, inasmuch as the results show only the proportions of the various ingredients and not their arrangement. Chemical analysis does, however, serve as a valuable means of detecting adulterations in cement, and also shows whether those elements believed to be harmful are present in too large quantities.

Cement, limestone, marl and clay are all composed of essentially the same elements, though in different proportions and in different combination. The latter statement can be better appreciated from the fact that although cement, limestone and marl are easily decomposed by hydrochloric acid, clay is scarcely affected at all. The various determinations made in the analysis of a cement are, therefore, practically the same as those made in the analysis of a limestone, marl, clay, slag or any other cement material, though in the case of the latter substances, an entirely different preliminary treatment is often necessary before these determinations can be made.

The analysis of any of the above-mentioned substances requires a considerable knowledge of analytical chemistry — a knowledge which can be acquired only by actual practice in the laboratory, and not from books alone. The chemical part of this manual has been written for the use of students who presumably have a knowledge of at least the rudiments of quantitative analysis.

The method for the analysis of cement hereafter given is, in general, the same as that reported by the New York section of the Society of Chemical Industry, and subsequently indorsed and published by a committee of the American Society of Civil Engineers. The authors have, however, somewhat enlarged upon the methods for the various determinations in the hope of making them more readily understood by less practiced chemists. Furthermore, in one or two places the method has been modified in such a way as to make it, in their estimation, far more satisfactory for general use. Alternate methods have been included for certain determ nations.

The methods of analysis given are for cement, and the raw material from which it is made, among which may be mentioned clay, limestone, marl, and slag. Of these materials clay alone is not at all decomposed by hydrochloric acid and hence must be subjected to a different treatment as described later. The analysis of a cement should consist of determinations of the following:

Loss on ignition	CaO.
Moisture.	MgO.
CO_2.	Alkalies.
SiO_2.	S.
Fe_2O_3.	SO_3.
Al_2O_3.	

Aside from these constituents, a cement always contains slight amounts of organic matter and sometimes also traces of manganese and phosphoric acid. The two latter, however, are so unusual the authors have not deemed it necessary to include methods for their determination. Average analyses of usual grades of cement have been given on p. 3, Chapter I.

Loss on ignition is always determined and represents the sum of CO_2, organic matter, and moisture. But the results are always somewhat affected by other factors as, for example, the changing of Fe_2O_3 to Fe_3O_4, of FeS to Fe_2O_3, and if manganese happens to be present, the changing of MnO to Mn_3O_4. Furthermore, small amounts of the alkalies are liable to be lost by volatilization during the ignition. With an ordinary cement, however, the error due to these factors is negligible.

Sulphur may be present in a cement as sulphate or as sulphide — both are usually present. The sulphur as sulphate is first determined, and then the total sulphur is determined as sulphate. From the difference between these two determinations the amount of sulphur existing as sulphide can be easily calculated.

Frequently CO_2 is not determined directly, but is calculated from the loss on ignition, and likewise many chemists determine the alkalies only by difference. Obviously this practice is far from good for very exact analyses and for this reason the authors have included methods for all determinations. For ordinary practical work, however, the calculation of CO_2 and of alkalies is probably entirely sufficient. In all analyses it is advisable to run duplicates, or "checks."

Preparation of the Sample for Analysis.

The sampling of cement has been discussed rather fully in a previous chapter (p. 7), but inasmuch as only a small

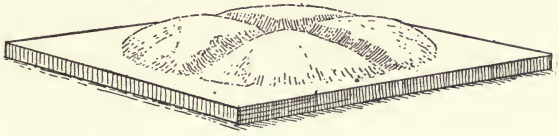

Fig. 37.

sample is employed in the chemical analysis, usually about 25 grams, a few words may be said as to the method employed in reducing the sample to this size. This is accomplished by "quartering." The sample is spread out on a large sheet of paper and divided into four equal parts by means of two lines drawn perpendicular to each other, as shown in Fig. 37. Two diagonally opposite quarters are then brushed away (Fig. 38), and the two remaining quarters are combined and thoroughly mixed. This is

again spread out and quartered, etc., the process being continued until about 25 grams are left.

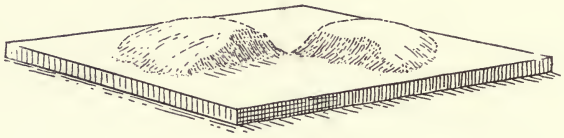

Fig. 38.

In sampling limestone, slag, clay, etc., it is not necessary to reduce the entire sample to fine powder. The material should be broken up to pea size and then quartered until about 100 grams are left. This 100 grams should then be finely powdered on a bucking board or in a mortar, and passed through a 100-mesh sieve. Of the finely powdered sample, 25 grams are sufficient.

The finished sample should be transferred to a tightly stoppered bottle or weighing flask, and should be properly labeled with number, date received, source and any other data which may be peculiar to the sample.

Analysis of Cement.

Loss on Ignition. (CO_2 + H_2O + organic matter).*

Weigh out into a platinum crucible about 0.5 gram of the finely divided sample. Place the crucible on a triangle and apply heat, gently at first, then with the full force of a good blast lamp for 15 minutes. The blast flame should never be directed vertically against the bottom of the

* See p. 82 for causes of error in this determination.

crucible but preferably at an angle against the side and bottom.

Cool the crucible in a dessicator and weigh. Heat again for 5 or 10 minutes in the blast flame, cool and weigh. Repeat until the weight is constant. The loss in weight is known as the " loss on ignition," and represents the sum of the CO_2, H_2O and organic matter.

Silica (SiO_2).

Carefully transfer the residue from the above determination of loss on ignition* to a platinum dish and carefully rinse out the crucible two or three times with dilute HCl, adding the rinsings, of course, to the contents of the dish. Add enough more dilute HCl to entirely cover the powder, place the dish on a water bath and evaporate to dryness — that is, until the odor of HCl can no longer be detected. Treat the residue with 10 c.c. strong HCl, digest on the water bath for 10 minutes with occasional stirring, and dilute with 35 c.c. of water. Allow the dish to digest on the water bath for 15 minutes. Filter through a small filter and wash the residue (SiO_2) thoroughly with hot water.

Transfer the filtrate to a platinum dish and again evaporate† to dryness on the water bath. Allow to stand on the

* A fresh sample may be used instead of taking the residue from the determination of loss on ignition. Such a procedure is certainly advisable if time is a factor.

† The second evaporation for silica is frequently omitted where great accuracy is not desired. The authors strongly advise two evaporations, however.

water bath until the odor of HCl is no longer perceptible. Moisten the residue with 5 c.c. strong HCl, digest for 10 minutes, and dilute with 35 c.c. of water. Allow to digest on the water bath for 10 or 15 minutes. Filter through a small filter and wash thoroughly with hot water. (The filtrate, *A*, is used for the determination of Fe_2O_3 and Al_2O_3.)

Transfer the two papers containing silica, while still wet, to a weighed platinum crucible, cover and apply heat, gently at first to dry and char the paper, then remove the cover and gradually increase the temperature to burn the paper. After the paper is burned, heat the crucible for 15 minutes over a good blast flame, being careful to prevent the blast from playing in or close to the mouth of the crucible. Cool in a desiccator and weigh. The process of heating, cooling and weighing should be continued until the weight is constant. This gives total silica (SiO_2).

The ignited silica should be white. If it is dark colored it is impure and may contain some particles which were not decomposed by the acid. In order to purify the silica, it must first be fused with sodium carbonate. Add to the silica in the crucible about five times its weight of pure, dry sodium carbonate and mix thoroughly by means of a platinum spatula or glass rod. Fuse over a good Bunsen burner as described later under the heading, "Analysis of Clay" (p. 107). After the fused mass has cooled, dissolve it in water, carefully add dilute HCl until in excess, and evaporate to dryness on the water bath. Take up the

silica as described above, etc. (The filtrate should be added to the main filtrate, *A*.)

Iron and Alumina ($Fe_2O_3 + Al_2O_3$).

To the filtrate, *A*, from the second evaporation for silica, add a little NH_4Cl solution and a few drops of strong HNO_3. Heat to boiling and precipitate the iron and alumina as hydrates by adding a slight excess of NH_4OH. Again heat to boiling for a moment,* wash once by decantation with boiling water containing a drop of NH_4OH, and filter as rapidly as possible. Wash the precipitate on the filter once or twice with boiling water containing a drop of NH_4OH. In this precipitation an effort should be made to keep the solution as hot as possible and to filter as quickly as possible. If allowed to cool, some alumina is changed to a soluble form and hence results in loss.† The filtration may be advantageously hurried by use of a force filter and platinum cone as suggested by Treadwell.‡ (The filtrate, *B*, is used for the determination of lime, CaO.)

Redissolve the precipitated $Fe(OH)_3$ and $Al(OH)_3$ on the filter by means of a small amount of hot dilute HCl, and allow the solution to run into a clean beaker. Wash the paper well with hot water. Heat to boiling and reprecipitate the iron and alumina by adding a slight excess of

* If boiled too long the precipitate becomes gelatinous and filtration is difficult.

† Alumina lost here will appear later in the precipitate of calcium and will be weighed as CaO.

‡ See "Analytical Chemistry," Treadwell-Hall.

NH_4OH as before. Quickly filter while hot and wash with hot water containing a drop of NH_4OH. (The filtrate, *C*, is added to filtrate *B* from the first precipitation of iron and alumina and is used for the determination of lime, CaO.)

Dry the filter paper containing the iron and alumina in a drying oven. When thoroughly dry fold the paper to a small volume and twist it onto the end of a platinum wire. Carefully burn the paper on the wire, holding it so that the ash will fall into a weighed platinum crucible. When the wire is cool, brush any adhering particles of oxide into the crucible. Place the crucible without cover on a triangle and ignite by means of a good Bunsen burner. Do not use the blast lamp. Cool and weigh. The combined weight of the Fe_2O_3 and the Al_2O_3 is thus obtained.*

Fe_2O_3.

To the crucible containing the Fe_2O_3 and Al_2O_3 add about 3 grams of $K_2S_2O_7$† and apply heat by means of a Bunsen burner turned low. Gradually increase the heat until the bottom of the crucible looks red as seen from above. Allow

* If the material under examination contains phosphoric acid, the latter will be present in the precipitate of iron and alumina and will be weighed as P_2O_5.

To determine the amount present, a separate sample should be employed, the phosphoric acid being precipitated from nitric acid solution by means of ammonium molybdate solution, and weighed as magnesium pyrophosphate, $Mg_2P_2O_7$, or as phosphomolybdic anhydride, 24 $MoO_3.P_2O_5$, as described in vol. ii, "Analytical Chemistry," by Treadwell-Hall.

The result is calculated to P_2O_5 and the amount subtracted from the total Fe_2O_3 and Al_2O_3 found.

† $KHSO_4$ is frequently employed instead of $K_2S_2O_7$. The latter salt is preferable.

to cool; then dissolve in water. The solution thus obtained contains the iron and alumina in the form of sulphates together with the excess of K_2SO_4.

To determine the Fe_2O_3, the solution is acidified with H_2SO_4, the iron reduced to the ferrous condition, and then titrated with a standardized $KMnO_4$ solution. The reduction of the iron may be brought about by one of the three methods described below.

Reduction by Means of Zinc. Introduce the solution of iron and alumina from the bisulphate fusion into a 200 c.c. Erlenmeyer flask fitted with a one-hole rubber stopper, carrying an exit tube as shown in the drawing (Fig. 39). Add about 3 grams of pure, finely divided zinc and a few cubic centimeters of concentrated H_2SO_4. Tightly stopper the flask and allow the outer end of the exit tube to dip into a beaker of water as illustrated. This serves as a trap. The hydrogen generated by the sulphuric acid and zinc completely reduces the iron to the ferrous condition and drives all air from the flask.

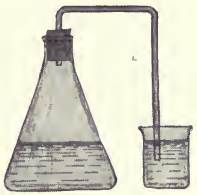

FIG. 39.

When the zinc is entirely dissolved, filter rapidly through a Gooch crucible and wash the crucible several times with small amounts of water. Remove the stopper and quickly titrate the solution in the filter flask with a standardized solution of $KMnO_4$. The number of cubic centimeters of $KMnO_4$ solution used, multiplied by its Fe_2O_3 factor, gives the total weight of Fe_2O_3.

Reduction with a Jones Reductor. A very convenient, as well as quick, method of reduction is accomplished by the aid of a Jones reductor. This apparatus is a glass tube of the form shown in Fig. 40, and is fitted to a filter flask by a tightly fitting rubber stopper. The reductor is filled as follows: Directly over the stopcock a few glass beads are placed and over these a little glass wool. This is then covered with a layer of sand. The balance of the tube, up to the enlargement at the top, is then filled with pure granulated zinc, after which the apparatus is ready for use. Once filled, it may be used indefinitely with no care save the occasional addition of a little zinc as the column of this metal is reduced by the action of acid. When not in use, the reductor should be tightly stoppered to prevent evaporation, and under no circumstances, either when in use or not in use, should the surface of the liquid be allowed to recede below the top of the column of zinc.

FIG. 40.

The operation of reduction is as follows: Suction is applied to the filter flask and the stopcock of the reductor is so regulated as to allow the liquid to be slowly drawn through the reductor into the filter flask. At first a little dilute H_2SO_4 is drawn through the apparatus. The liquid to be reduced, which should contain enough H_2SO_4 to have a moderate action on zinc, is then run through, and this is

followed by successive small quantities of water to displace all acid. The reductor is then removed from the flask and the solution in the latter is rapidly titrated to a permanent pink color by means of standardized $KMnO_4$ solution. As before mentioned, great care must be exercised during the process of reduction to prevent the surface of the liquid from falling below the top of the column of zinc.

Reduction by Means of H_2S. The solution from the bisulphate fusion is placed in a 200 c.c. flask, tightly stoppered with a rubber stopper provided with two tubes through which gas can enter and leave the flask (Fig. 41). The tube through which the gas enters should dip below the surface of the liquid, as is shown in the drawing. The solution is heated to boiling and a current of pure H_2S is passed through until the solution is saturated. The tube through which the gas enters is now connected with a CO_2 generator, and while the solution in the flask is still boiling CO_2 is passed through until all H_2S has been removed — until wet lead acetate paper held near the exit tube is no longer blackened. The solution is then allowed to cool in the atmosphere of CO_2, after which it is filtered and then titrated with standard $KMnO_4$ as previously described.

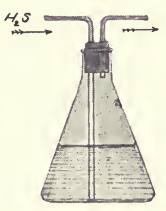

FIG. 41.

Al_2O_3.

The total weight of Al_2O_3 is simply the difference between the combined weight of Fe_2O_3 and Al_2O_3, and Fe_2O_3 alone.

Lime (CaO).

Combine the filtrates *B* and *C* from the two precipitations of iron and alumina, acidify with HCl and evaporate to a volume of about 250 c.c. Make alkaline with NH_4OH and heat to boiling.* While the solution is still boiling, add an excess of a boiling solution of $(NH_4)_2C_2O_4$. Boil for a moment longer and then allow to stand and settle for 20 minutes, or, better still, over night. Filter and wash thoroughly with hot water containing a little $(NH_4)_2C_2O_4$.† (The filtrate, *D*, is used for the determination of magnesia, MgO.)

Place the filter paper containing the precipitate while still wet in a weighed platinum crucible and heat carefully in the Bunsen flame until the paper is burned. Cover the crucible and ignite for 15 or 20 minutes in a good strong blast, to completely reduce the oxalate to oxide. After ignition quickly transfer the hot crucible to a desiccator, preferably one fitted with a U-tube containing soda lime as shown in Fig. 42. When cool, weigh. The crucible should be reheated, cooled and weighed until the weight is constant. This gives total lime, CaO.

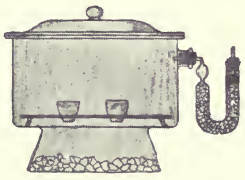

Fig. 42.

* A little $Al(OH)_3$ often separates at this point. This will not happen, however, if the $Fe(OH)_3$ and $Al(OH)_3$ precipitates were kept hot while being filtered. If any separates, it should be filtered off, ignited and weighed.

† Some chemists prefer to redissolve and reprecipitate the calcium, but this is entirely unnecessary in ordinary analytical work.

Magnesia (MgO).

To the filtrate *D* from the calcium oxalate precipitation, add HCl until acid. Evaporate to a volume of about 75 or 100 c.c. Then add an excess of Na_2HPO_4 or $NaNH_4HPO_4$ solution and ammonia in moderate excess. Allow to stand at least 4 hours (better over night).* Filter and wash once or twice with a 2.5 per cent solution of NH_4OH.

Dissolve the precipitate on the paper in a little hot, dilute HNO_3. Add a few drops of Na_2HPO_4 solution and then NH_4OH, drop by drop, until in moderate excess, with constant agitation of the solution. Allow to stand 4 hours, or over night if convenient. Filter and wash with 2.5 per cent NH_4OH. Reject the filtrate.

Transfer the precipitate and paper while still wet to a weighed platinum crucible and heat with a low Bunsen flame to dry and char the paper. Burn the paper at as low a temperature as possible. When entirely burned, ignite the crucible in a weak blast flame, cool and weigh. Repeat until constant weight is obtained. This gives weight of MgO as $Mg_2P_2O_7$. This weight, multiplied by the factor 0.3621, gives total magnesia, MgO.

Sulphuric Acid (SO_3).

Into a porcelain evaporating dish or casserole, weigh out one gram of the finely powdered sample, add dilute HCl to

* It is not necessary to remove ammonium salts before making this first precipitation of magnesia. Large amounts of ammonium salts do, however, greatly retard precipitation.

cover the powder and take to dryness on the water bath. (By this treatment all the sulphur which was present in the cement as sulphide will be expelled as H_2S.) Dissolve the residue in water,* treat with NH_4OH and $(NH_4)_2CO_3$ in excess, filter and wash thoroughly. Concentrate the filtrate, acidify with HCl, heat to boiling for a moment and precipitate the sulphuric acid as $BaSO_4$ by adding a boiling solution of $BaCl_2$, drop by drop, with constant stirring, until in excess. Allow to stand on the water bath about two hours, filter and wash. Or, if convenient, allow to stand over night at the ordinary temperature before filtering. Transfer the filter and precipitate to a platinum crucible and burn the paper at as low a temperature as possible. Ignite for 10 or 15 minutes with the cover off, cool and weigh as $BaSO_4$. The weight of $BaSO_4$ obtained multiplied by the factor 0.3430 gives weight of sulphuric acid (SO_3).

Total Sulphur.

For the determination of total sulphur, two methods are available. The first to be described below, the fusion method, is the one reported by the committee on uniform methods of analysis. The alternate method is a very good one and is especially good for the determination of sulphur in cement.

* Many chemists filter the solution at this point and precipitate the sulphuric acid immediately without going through the NH_4OH and $(NH_4)_2CO_3$ treatment. This procedure seems to be very satisfactory though it is liable to give too high results, inasmuch as the $BaSO_4$ brings down other substances mechanically.

Fusion Method. About 1 gram of the powdered sample is weighed out into a large platinum crucible and about 5 or 6 grams of pure dry Na_2CO_3 (free from sulphur) and 0.3 gram of KNO_3 are added.* This charge should be intimately mixed by means of a platinum spatula or glass rod, and then fused. In order to protect the contents of the crucible from sulphur in the flame, the crucible should be placed through a hole in a piece of asbestos board held in a slightly inclined position. A good Bunsen burner is sufficient to bring about the fusion. Heat until a quiet fusion is obtained.

After cooling, the melt is treated in the crucible with boiling water and the solution poured into a tall beaker. More hot water is added and the whole is agitated until disintegration is complete. The solution is then filtered, the filtrate is acidified with HCl and diluted to about 250 c.c. It is then heated to boiling and the SO_3 is precipitated as $BaSO_4$, by the addition, drop by drop, of a boiling solution of $BaCl_2$ as previously described in the determination of sulphuric acid. The precipitate is filtered, washed, ignited and weighed as described in the same place. The weight of $BaSO_4$ obtained, multiplied by the factor 0.1374, gives total sulphur.

Bromine Method. This method depends upon the oxidation of S to SO_3 by means of Br water. Weigh out 1 gram

* As the reagents here used, Na_2CO_3 and KNO_3, are liable to contain sulphur, a blank should be run first. Results of subsequent analyses can then be corrected for the amount found.

of the sample into a porcelain evaporating dish or casserole and treat with Br water in excess. Allow to digest on the water bath for 10 or 15 minutes, then acidify with HCl and evaporate to dryness. Moisten the residue with 1 c.c. of concentrated HCl and dilute with 200 c.c. of water. This solution is then treated with NH_4OH and $(NH_4)_2CO_3$, etc., as described previously in the determination of sulphuric acid (SO_3). This result gives all the sulphur as $BaSO_4$; this figure, multiplied by the factor 0.1374, gives total sulphur.

Sulphur Existing in the Cement as Sulphide.

From the weight of $BaSO_4$, obtained in the determination of total sulphur, subtract the weight of $BaSO_4$ obtained in the determination of sulphuric acid (SO_3). The difference will represent the sulphur existing in the cement as sulphide, weighed in the form of $BaSO_4$. By multiplying this weight by the factor 0.1374 the weight of sulphur (S) existing as sulphide will be ascertained.

Moisture.

If a determination of moisture is desired, it can be made in the following simple manner. Weigh out into a platinum crucible 1 or 2 grams of the sample and heat for an hour in an air bath maintained at a temperature of 110°. The crucible is then cooled in a desiccator and weighed. This procedure should be repeated until the weight is con-

stant. The loss in weight represents moisture in the cement.*

Alkalies.

In the analysis of cement, many chemists determine alkalies solely by difference, a procedure which is entirely satisfactory for all ordinary purposes. In accurate work, however, and especially in the analysis of clay, the alkalies must be determined gravimetrically. For this determination the J. Lawrence Smith method alone is satisfactory.

About 0.5 gram of the sample is intimately mixed with an equal weight of pure NH_4Cl and 3 grams of pure $CaCO_3$. This mixing is usually done in an agate mortar. The mixture is transferred to the crucible and covered with about 1 gram of pure $CaCO_3$.

Fig. 43.

The J. Lawrence Smith crucible, which was especially designed for this determination, is a long tube-like receptacle with a cap for the open end. When the crucible is filled it is capped and placed in an inclined position in a clay cylinder, as shown in Fig. 43. The outer end of the crucible remains cool and thus prevents loss by volatilization. Instead of a clay cylinder a piece of asbestos

* A rough determination of moisture is sometimes made in testing laboratories, with a 100 or 200 gram sample in a porcelain evaporating dish, the weighings being made with an ordinary scale as described on page 11. Such a determination is probably entirely satisfactory for all practical purposes.

board with a hole in the center to admit the crucible may be conveniently used by clamping it in a vertical position.

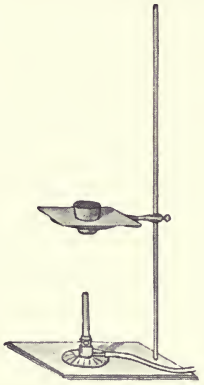

Fig. 44.

An ordinary platinum crucible of 25 or 30 c.c. capacity may be used for this determination with equally good results, though it requires a little more care during the process of heating. If an ordinary crucible is employed, it should be fitted into a hole in a piece of asbestos board so that about half or two-thirds of the crucible protrudes below the board (Fig. 44). The crucible should be covered with a platinum lid.

Heat is at first applied by means of a Bunsen burner turned very low, or placed some distance below the crucible. If a regular J. Lawrence Smith crucible is employed, a flat flame should be used. When the odor of ammonia is no longer perceptible, the temperature is gradually raised until the full flame of a strong Bunsen burner is employed. Two burners can be used with the J. Lawrence Smith crucible. This heat is continued for about 40 minutes. After the crucible is cool, the cintered mass is loosened by gently tapping the crucible and is then transferred to a porcelain or platinum dish, and treated with 50–75 c.c. of water. Any of the mass which sticks to the crucible is loosened by digesting with a little water, and washed into the dish.

The dish containing the cintered mass and water is

digested on the water bath for 30 minutes and any large particles are broken by means of a glass rod. Water lost by evaporation should be replaced.

When disintegration is complete, the solution in the dish is decanted through a filter and the residue washed 3 or 4 times by decantation. The residue is then transferred to the filter and washed until free from chlorides.*

The filtrate is then treated with an excess of NH_4OH and $(NH_4)_2CO_3$, heated to boiling, filtered and washed once or twice. Inasmuch as the precipitate probably still contains traces of alkalies, it should be dissolved in HCl and reprecipitated by means of NH_4OH and $(NH_4)_2CO_3$ in excess. It is now filtered and washed thoroughly. The two filtrates are combined, evaporated to dryness on the water bath and then gently ignited to expel all ammonium salts. Dissolve the residue in a small amount of water and treat with a little NH_4OH and $(NH_4)_2C_2O_4$ to remove the last traces of Ca. Allow to stand over night; filter and wash, allowing the filtrate and washings to run into a weighed platinum dish or large crucible. Evaporate to dryness and ignite to expel ammonium salts as before. Allow to cool; moisten with HCl to change any carbonate into chloride, again evaporate to dryness and ignite at a low red heat. Cool in a desiccator and weigh. This gives total alkali as chloride.

Determination of Potassium (K_2O). Dissolve the ignited chlorides in a small amount of water and treat with a slight

* The residue on the filter should be entirely soluble in HCl.

excess of platinum chloride solution. The approximate amount of platinum chloride solution necessary can be easily calculated from the weight of the alkali as chloride determined above. The solution should be so dilute that the precipitate redissolves when heated on the water bath. Allow to evaporate on the water bath until the residue solidifies on cooling. Drench the solidified mass with absolute alcohol or with 80 per cent alcohol, and decant the liquid through a very small filter. Wash by decantation with alcohol of the same strength, being careful to bring as little as possible of the precipitate on the paper. The dish and filter are allowed to dry for a few moments, after which the contents of the dish are transferred to a weighed platinum crucible. Any particles of the K_2PtCl_6 precipitate still adhering to the dish should now be washed through the filter by means of hot water, the solution being caught in the crucible. The latter is then placed on the water bath for a time and then heated for a few moments in an air bath at 135°. The dish should be covered during the first few moments in the air bath to prevent loss due to decrepitation. The crucible is cooled in a desiccator and weighed. The weight of K_2PtCl_6 thus obtained, multiplied by the factor 0.1937, gives the weight of potassium as K_2O.

Determination of Sodium Oxide (Na_2O). To calculate the weight of Na_2O, multiply the weight of K_2PtCl_6 above found by the factor 0.3067 to find the weight of potassium as KCl. This weight should be subtracted from the total weight of alkalies as chloride previously determined. The

difference, which represents the weight of sodium as NaCl, should be multiplied by the factor 0.5303, which gives the weight of sodium as Na_2O.

Carbon Dioxide (CO_2).

The determination of CO_2 may be made by either of two totally different methods, the " indirect " and the " direct," both of which are described below.

The " indirect " method has the advantage of being rapid and requires less apparatus. It is not as accurate as the direct method, although for determinations in limestone, marl, or other substances rich in CO_2, it yields very satisfactory results. This method is far less satisfactory for cement, inasmuch as the percentage of CO_2 therein is very small.

The " direct " method is very accurate and especially well adapted to substances containing only small amounts of CO_2. The apparatus required, although somewhat complicated, is easily assimilated and consists only of such pieces as are ordinarily found in a chemical laboratory.

Indirect Method.

The principle involved in the " indirect " method is the determination of the loss in weight due to expulsion of the CO_2. A special form of apparatus is needed for this determination, the Schrötter apparatus,* shown in Fig. 45, being one of the more common forms.

* Many other forms of apparatus have been described for this determination, among them those of Bunsen, Fresenius, Mohr, Geissler,

Procedure. The apparatus is first cleaned and dried. Compartment *b* is then about one-third filled with concentrated H_2SO_4 and compartment *c* is nearly filled with dilute HCl (1 to 3). The glass stopper is then replaced in *c* and the end of *b* is closed by means of a piece of rubber tubing carrying a piece of glass rod in one end. The apparatus is then weighed.

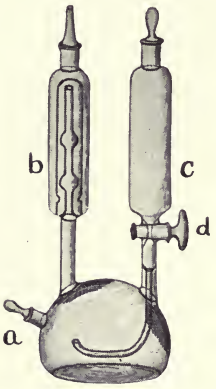

FIG. 45.

The sample is then introduced into the apparatus through *a*, after which the stopper is quickly replaced and the apparatus is again weighed. The increase in weight is the weight of the sample.

The rubber stopper is now removed from the end of *b* and stopcock *d* is partially opened to allow the HCl in *c* to run slowly down into the flask and come into contact with the sample. The CO_2 is thus liberated and is forced out of the apparatus through *b*, the concentrated H_2SO_4 in this compartment preventing the escape of moisture. The flow of HCl is so regulated that the CO_2 bubbles through the H_2SO_4 slowly — not faster than two bubbles per second.

After decomposition is complete and all HCl has run from *c* into the flask, stopcock *d* is closed and the flask is gradually

Rohrbeck, etc. All of these are designed on the same principle as the Schrötter apparatus and all are equally well adapted to the determination of CO_2 in carbonates, etc.

heated until the boiling point is reached, in order to expel any CO_2 remaining in solution.

The stopcock *d* is then quickly opened and the stopper in *c* is quickly replaced by a one-hole rubber stopper carrying a drying tube filled with soda lime. The end of compartment *b* is now connected with a $CaCl_2$ tube which in turn is connected with an aspirator and a current of air is slowly drawn through the apparatus to drive out all CO_2.

After several liters of air have been drawn through the apparatus, and the liquid contents of the latter are cool, the glass stopper is again placed in *c*, and the end of *b* is again closed by means of the rubber tube and glass rod. The apparatus is now carefully weighed. The loss in weight represents the weight of CO_2.

Direct Method.

In the direct determination of CO_2, the gas, evolved by the action of acid, is first passed through a number of drying tubes, after which it is absorbed in soda lime or a concentrated solution of KOH. By weighing the absorption tubes before and after the experiment, the actual weight of the CO_2 is obtained.

The apparatus employed is shown in Fig. 46. The flask *b* is of about 100 c.c. capacity and is used for the decomposition of the sample. The U-tube *c* contains a few glass beads moistened with a little concentrated H_2SO_4.* The

* A small condenser can be advantageously employed between *b* and *c*, so arranged as to cause moisture condensed to run back into *b*.

first half of *d* is filled with $CaCl_2$ and the second half with anhydrous $CuSO_4$ and glass wool or pumice. *e* contains

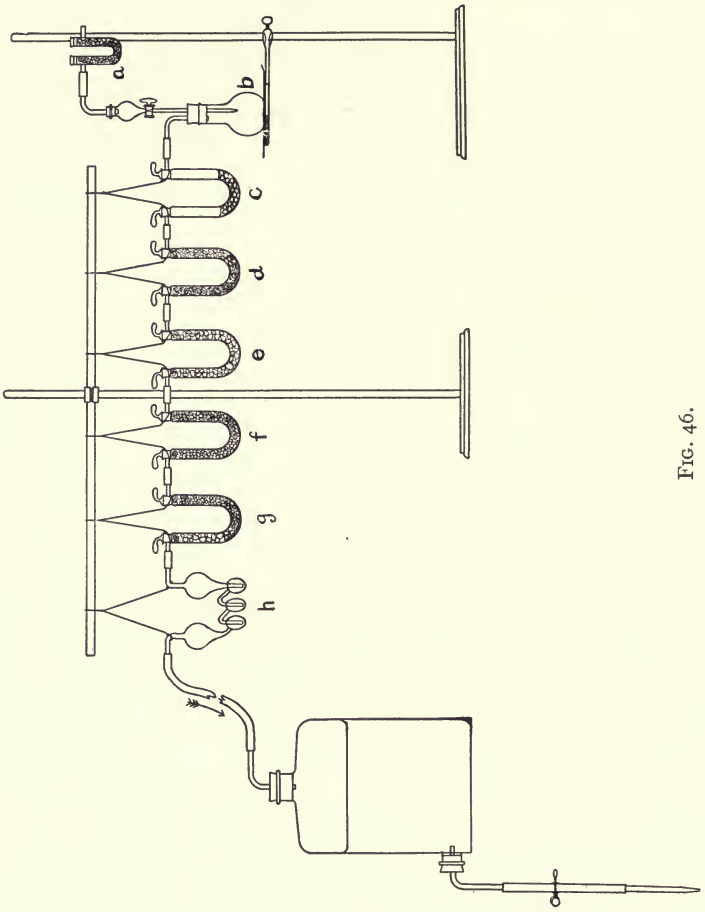

FIG. 46.

$CaCl_2$ to remove final traces of moisture. Glass stoppered U-tubes *f* and *g* are the absorption tubes. The former is

filled with soda lime and the latter contains soda lime in the first half and $CaCl_2$ in the other half. This $CaCl_2$ absorbs any moisture liberated by the absorption of CO_2 in the soda lime. *h* is a Liebig bulb filled with concentrated H_2SO_4. This prevents moisture from backing up into the absorption tubes, and also indicates the rate at which the gas is passing through the apparatus. The bulb *h* is connected directly to the aspirator. The small U-tube *a* is filled with soda lime, and is attached as shown in the figure while air is being drawn through the apparatus, thus freeing it from CO_2.

After filling the various tubes as described above, they are joined together as shown in the drawing, by means of small pieces of rubber tubing. Every joint should be securely wired.

Procedure. Air is first passed through the apparatus for some time in order to free it from traces of CO_2. During this process *h* may be connected directly to *e*, as it is not necessary for the absorption tubes to be in position. The weighed sample is then introduced into the flask *b*, which is immediately closed. The tube *a* is now removed and the funnel tube in *b* is filled with dilute HCl (about 1 : 3). The two absorption tubes, which should be tightly closed, are now weighed, after which they are connected with the apparatus, as shown, and the joints tightly wired.

The dilute HCl in the funnel tube is now allowed to run into *b* a little at a time, to decompose the carbonate. The rate of flow must be so regulated that not more than two bubbles a second pass through *h*. When all HCl has run

into *b*, the stopcock in the funnel tube is closed and the contents of the flask are gradually heated to the boiling point to drive out any CO_2 in solution in the acid. The flame is then removed, the stopcock in the funnel tube is opened and the U-tube *a* connected as shown in the drawing. By means of the aspirator, air is now drawn through the apparatus to completely sweep all CO_2 into the absorption tubes.

After several liters of air have been aspirated through the apparatus, the stoppers in *f* and *g* are tightly closed, after which they are taken from the apparatus and weighed. The increase in weight is the weight of the CO_2.

Analysis of Limestone.

Limestone is essentially a carbonate of calcium, but it always contains more or less silica, iron, alumina, and magnesia as impurities. Limestone is very readily attacked by dilute HCl, hence no preliminary treatment is necessary to prepare it for the various determinations.

Procedure. Weigh out 0.5 gram of the finely powdered sample into a good-sized platinum dish and add water to cover the powder. Add dilute HCl, a little at a time, being careful to cover the dish quickly with a watch glass after each addition. When sufficient acid has been added and effervescence no longer takes place, wash the cover glass, allowing the washings to run into the dish, place the latter on the water bath and evaporate to dryness. Take up with HCl and water, etc., and proceed with the

various determinations as heretofore described under the heading, "Analysis of Cement."

Moisture, loss on ignition, and CO_2 are made with separate samples. Frequently CO_2 is determined by difference — a method which yields results entirely satisfactory for all ordinary purposes.

Determinations of SO_3, total S, and alkalies are not necessary.

Analysis of Marl.

In the analysis of marl, decomposition is effected by means of HCl as described under the heading, "Analysis of Limestone." Marl may, however, contain more or less clay which is not decomposed by the acid. If this is the case, as can readily be told from the appearance of the silica, the latter must be fused with dry Na_2CO_3, as described under the heading, "Analysis of Clay."

In the analysis of marl the same determinations should be made as in the case of cement — the analysis should always include determinations of S, SO_3 and alkalies.

Analysis of Slag.

In the analysis of slag, proceed exactly as in the case of cement, making all the determinations as described under the heading, "Analysis of Cement."

Analysis of Clay.

In the analysis of clay, practically the same determinations are made as in the case of cement, although a far

different preliminary treatment is necessary on account of the insolubility of clay in HCl. This preliminary treatment consists in fusing the clay with Na_2CO_3, thereby changing its ingredients into forms which are readily decomposed by HCl.

About 0.5 gram of the finely powdered sample* is weighed out into a platinum crucible of 25 or 30 c.c. capacity, 3 or 4 grams of pure dry Na_2CO_3 are added, and the whole is intimately mixed by stirring with a platinum spatula or glass rod. A little Na_2CO_3 is now sprinkled on top, the crucible covered, placed on a triangle and heated. Heat should be applied gently at first by means of a Bunsen burner turned low. The temperature is then gradually raised until the full force of a Teclu burner or blast lamp is used. The heating is continued until CO_2 is no longer evolved, and the contents of the dish are in a state of quiet fusion.

When the crucible is cool it is placed in a beaker and partially covered with water. It is then allowed to digest until the melt is thoroughly disintegrated. The crucible is then withdrawn and washed with water and a little dilute HCl, the washings being added to the contents of the beaker.

The beaker is then covered with a watch glass and HCl is added a little at a time, the watch glass being quickly replaced after each addition of acid. When CO_2 is no

* It is customary to dry the sample at 105 or 110° in the air bath before making the analysis.

longer evolved, the contents of the beaker are transferred to a platinum dish and evaporated to dryness on the water bath. The various determinations SiO_2, Fe_2O_3, etc., are then made as heretofore described in the analysis of cement.

For the determination of alkalies in clay, exactly the same procedure is employed as for the determination of alkalies in cement.

APPENDIX

STANDARD SPECIFICATIONS AND UNIFORM METHODS OF TESTING AND ANALYSIS FOR PORTLAND CEMENT

EMBRACING

THE REPORT OF THE COMMITTEE ON STANDARD SPECIFICATIONS FOR CEMENT OF THE AMERICAN SOCIETY FOR TESTING MATERIALS; THE REPORT OF THE COMMITTEE ON UNIFORM TESTS OF CEMENT OF THE AMERICAN SOCIETY OF CIVIL ENGINEERS; AND THE REPORT OF THE COMMITTEE ON UNIFORMITY IN TECHNICAL ANALYSIS FOR LIMESTONES, RAW MIXTURES AND PORTLAND CEMENTS OF THE SOCIETY FOR CHEMICAL INDUSTRY (NEW YORK SECTION)

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APPENDIX.

STANDARD SPECIFICATIONS FOR PORTLAND CEMENT

Adopted by the American Society for Testing Materials, August 16th, 1909.

GENERAL OBSERVATIONS.

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.

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.

Specific gravity is useful in detecting adulteration. 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.

The sieves should be kept thoroughly dry.

Time of Setting.

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.

Constancy of Volume.

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.

In making the pats the greatest care should be exercised to avoid initial strains due to molding 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.

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, using a new sample. 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.

SPECIFICATIONS.

GENERAL CONDITIONS.

All cement shall be inspected.

Cement may be inspected either at the place of manufacture or on the work.

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.

The cement shall be stored in such a manner as to permit easy access for proper inspection and identification of each shipment.

Every facility shall be provided by the contractor and a period of at least twelve days allowed for the inspection and necessary tests.

Cement shall be delivered in suitable packages with the brand and name of manufacturer plainly marked thereon.

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.

Cement failing to meet the seven-day requirements may be held awaiting the results of the twenty-eight day tests before rejection.

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, and January 15, 1908, with all subsequent amendments thereto.

The acceptance or rejection shall be based on the following requirements:

PORTLAND CEMENT.

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 per cent has been made subsequent to calcination.

Specific Gravity.

The specific gravity of cement shall not be less than 3.10. Should the test of cement as received fall below this requirement, a second test may be made upon a sample ignited at a low red heat. The loss in weight of the ignited cement shall not exceed 4 per cent.

Fineness.

It shall leave by weight a residue of not more than 8 per cent on the No. 100, and not more than 25 per cent on the No. 200 sieve.

Time of Setting.

It shall not develop initial set in less than thirty minutes; and must develop hard set in not less than one hour. nor more than ten hours.

Tensile Strength

The minimum requirements for tensile strength for briquettes one square inch in cross section shall be as follows and the cement shall show no retrogression in strength within the periods specified:

Age.	*Neat Cement.*	*Strength.*
24 hours in moist air		175 lbs.
7 days (1 day in moist air, 6 days in water)		500 lbs.
28 days (1 day in moist air, 27 days in water)		600 lbs.
	One Part Cement, Three Parts Standard Ottawa Sand.	
7 days (1 day in moist air, 6 days in water)		200 lbs.
28 days (1 day in moist air, 27 days in water)		275 lbs.

Constancy of Volume.

Pats of neat cement about three inches in diameter, one-half inch thick at the center, 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° F. 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.

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.

The cement shall not contain more than 1.75 per cent of anhydrous sulphuric acid (SO_3), nor more than 4 per cent of magnesia (MgO).

REPORT OF COMMITTEE ON UNIFORM TESTS OF CEMENT OF THE AMERICAN SOCIETY OF CIVIL ENGINEERS.

Presented at the Annual Meeting, January 18th, 1911.

Your Committee on Uniform Tests of Cement presents the following report:

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. — 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.

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 *Engineering News*, Vol. 50, p. 60, 1903; and in *The Engineering Record*, Vol. 48, p. 49, 1903, is recommended.

Specific Gravity.

8. — *Significance.* — The specific gravity of cement is lowered by 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. — *Apparatus and Method.* — The determination of specific gravity is most conveniently made with Le Chatelier's apparatus.

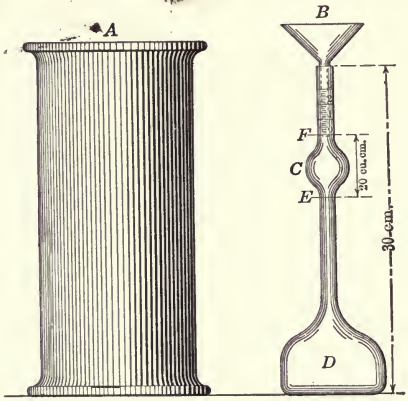


Fig. 1.

This consists of a flask (*D*), Fig. 1, of 120 cu. cm. (7.32 cu. in.) capacity, the neck of which is about 20 cm. (7.87 in.) 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. in.). The neck has a diameter of about 9 mm. (0.35 in.), and is graduated into tenths of cubic centimeters above the mark (*F*).

11. — Benzine (62° Baumé naphtha), or kerosene free from water, should be used in making the determination.

12. — The specific gravity is determined as follows:

The flask is filled with either of these liquids to the lower mark (*E*), and 64 gms. (2.25 oz.) of powder, cooled to the temperature of

the liquid, is gradually introduced through the funnel (*B*) [the stem of which extends into the flask at the top of the bulb (*C*)], until all 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 gms. of the powder.

13. — The specific gravity is then obtained from the formula:

$$\text{Specific Gravity} = \frac{\text{Weight of Cement, in grams}}{\text{Displaced Volume, in cubic centimeters}}.$$

14. — 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. The determination of specific gravity should be made on the cement as received; and, should it fall below 3.10, a second determination should be made on the sample ignited at a low red heat.

15. — 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 removed in a similar manner by pouring into the flask a small quantity of clean liquid benzine or kerosene and repeating the operation.

Fineness.

16. — *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.

17. — 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.

18. — *Apparatus.* — The sieves should be circular, about 20 cm. (7.87 in.) in diameter, 6 cm. (2.36 in.) high, and provided with a pan, 5 cm. (1.97 in.) deep, and a cover.

19. — The wire cloth should be of brass wire having the following diameters:

No. 100, 0.0045 in.; No. 200, 0.0024 in.

20. — 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.

21. — Fifty grams (1.76 oz.) or 100 g. (3.52 oz.) should be used for the test, and dried at a temperature of 100° Cent. (212° Fahr.) prior to sieving.

22. — *Method.* — 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 steel shot. The results should be reported to the nearest tenth of 1 per cent.

Normal Consistency.

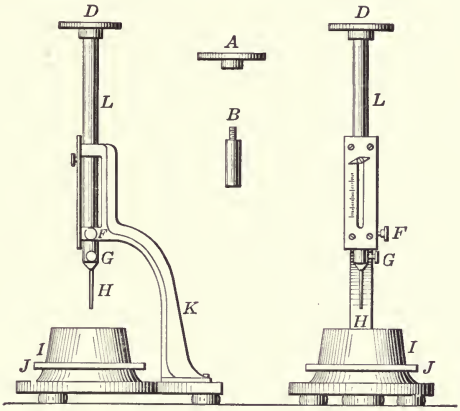
23. — *Significance.* — The use of a proper percentage of water in making the pastes * from which pats, tests of setting, and briquettes are made, is exceedingly important, and affects vitally the results obtained.

24. — 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.

25. — The Committee recommends the following method for determining normal consistency.

* 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.

26. — *Method, Vicat Needle Apparatus.* — This consists of a frame (*K*), Fig. 2, bearing a movable rod (*L*), with the 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 gms. (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*), 7 cm. (2.76 in.) in diameter at the base, 4 cm. (1.57 in.) high, resting on a glass plate (*J*), about 10 cm. (3.94 in.) square.



VICAT NEEDLE.

FIG. 2.

27. — 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 gms., is kneaded into a paste, as described in Paragraph 52, 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 in. 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, resting on the plate, is placed under the rod bearing the cylinder, which is brought in contact with the surface and quickly released.

28. — The paste is of normal consistency when the cylinder in one minute from the time it is released 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. The apparatus must be free from all vibrations during the test.

29. — The trial pastes are made with varying percentages of water until the correct consistency is obtained.

30. — 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.

31. — 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 the table below.

PERCENTAGE OF WATER FOR STANDARD MORTARS

Neat.	One cement, three standard Ottawa sand.	Neat.	One cement, three standard Ottawa sand.	Neat.	One cement, three standard Ottawa sand.
15	8.0	23	9.3	31	10.7
16	8.2	24	9.5	32	10.8
17	8.3	25	9.7	33	11.0
18	8.5	26	9.8	34	11.2
19	8.7	27	10.0	35	11.5
20	8.8	28	10.2	36	11.5
21	9.0	29	10.3	37	11.7
22	9.2	30	10.5	38	11.8

Time of Setting.

32. — *Significance.* — The object of this test is to determine the time which elapses 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 com-

plete the operation of mixing and moulding or incorporating the mortar into the work before the cement begins to set.

33. — It is usual to measure arbitrarily the beginning and end of the setting by the penetration of weighted wires of given diameters.

34. — *Method.* — For this purpose the Vicat Needle, which has already been described in Paragraph 26, should be used.

35. — In making the test, a paste of normal consistency is molded and placed under the rod (*L*), Fig. 2, as described in Paragraph 27; 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 gms. (10.58 oz.). The needle is then carefully brought in contact with the surface of the paste and quickly released.

36. — 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.

37. — 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.

38. — 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.

39. — 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 kneading the paste receives.

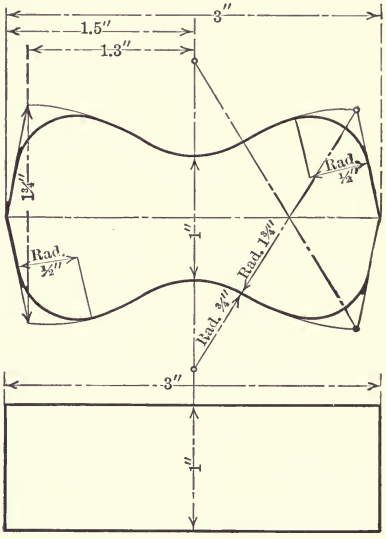
Standard Sand.

40. — 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 1 per cent passes a No. 30 sieve after one minute's continuous sifting of a 500-g sample.*

FORM OF TEST PIECES.

41. — For tension tests the Committee recommends the form of test piece shown in Fig. 3.



DETAILS FOR BRIQUETTE.

FIG. 3.

42. — For compression tests a 2-in. cube is recommended.

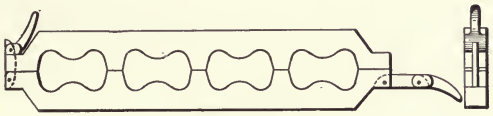
MOLDS.

43. — The molds should be made of brass, bronze, or some equally non-corrodible material, having sufficient metal in the sides to prevent spreading during molding.

44. — Gang molds, which permit molding a number of briquettes at one time, are preferred by many to single molds; since the

* This sand may be obtained from the Ottawa Silica Company at a cost of two cents per pound, f. o. b. cars, Ottawa, Illinois.

greater quantity of mortar that can be mixed tends to produce greater uniformity in the results. The type shown in Fig. 4 is recommended.



DETAILS FOR GANG MOULD.

FIG. 4.

45. — The molds should be wiped with an oily cloth before using.

MIXING.

46. — All proportions should be stated by weight; the quantity of water to be used should be stated as a percentage of the dry material.

47. — The metric system is recommended because of the convenient relation of the gram and the cubic centimeter.

48. — The temperature of the room and the mixing water should be as near 21° Cent. (70° Fahr.) as it is practicable to maintain it.

49. — 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.

50. — The quantity of material to be mixed at one time depends on the number of test pieces to be made; about 1,000 gms. (35.28 oz.) makes a convenient quantity to mix, especially by hand methods.

51. — 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 is no means of ascertaining when the mixing is complete without stopping the machine; and (3) the difficulty of keeping the machine clean.

52. — *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 one minute, 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.

Molding.

53. — Having worked the paste or mortar to the proper consistency, it is at once placed in the molds by hand.

54. — The Committee has been unable to secure satisfactory results with the present molding machines; the operation of machine moulding is very slow, and the present types permit of molding but one briquette at a time, and are not practicable with the pastes or mortars herein recommended.

55. — *Method.* — The molds should be filled immediately after the mixing is completed, the material pressed in firmly with the fingers and smoothed off with a trowel without mechanical ramming; the material should be heaped up on the upper surface of the mold, and, in smoothing off, the trowel should be drawn over the mold in such a manner as to exert a moderate pressure on the excess material. The mold should be turned over and the operation repeated.

56. — A check upon the uniformity of the mixing and molding 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.

57. — During the first 24 hours after molding, the test pieces should be kept in moist air to prevent them from drying out.

58. — A moist closet or chamber is so easily devised that the use of the damp cloth should be abandoned. 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

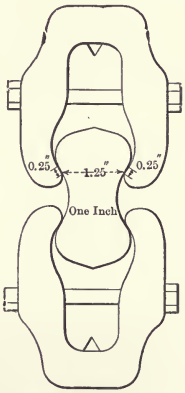
is not available, a cloth may be used and kept uniformly wet by immersing the ends in water. It should be kept from direct contact with the test pieces by means of a wire screen or some similar arrangement.

59. — 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.

60. — 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.

61. — The tests may be made on any machine. A solid metal clip, as shown in Fig. 5, 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 $\frac{1}{4}$ in. wide and the distance between the center of contact on the same clip should be $1\frac{1}{4}$ in.



FORM OF CLIP
Fig. 5.

62. — 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 briquettes 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 lb. per min. 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.

63. — *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.

64. — *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. (113° 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.

65. — For these tests, pats, about $7\frac{1}{2}$ cm. (2.95 in.) in diameter, $1\frac{1}{4}$ 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 in.) square], from cement paste of normal consistency.

66. — *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, after 24 hours in moist air, is maintained in air at ordinary temperature and observed at intervals.

67. — *Accelerated Tests.* — A pat is placed in an atmosphere of steam upon a wire screen 1 in. above boiling water for five (5) hours. The apparatus should be so constructed as to permit the free escape of steam and maintain atmospheric pressure. Since the type of apparatus used has a great influence on the uniformity of the results, that shown in Fig. 8 is recommended.

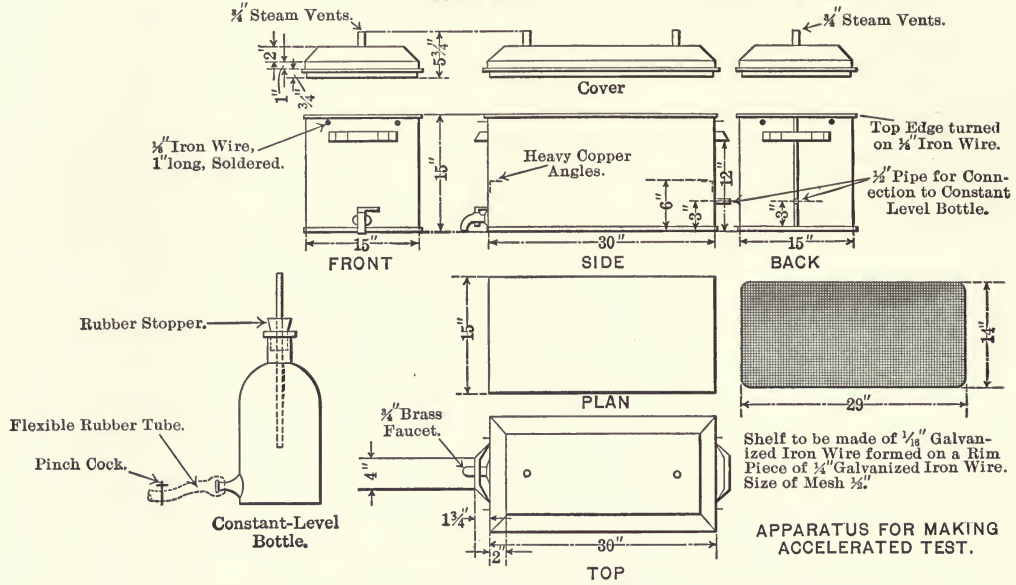
68. — To pass these tests satisfactorily, the pats should remain firm and hard, and show no signs of cracking, distortion or disintegration.

69. — 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.

70. — In the present state of our knowledge it cannot be said that cement should necessarily be condemned simply for failure to

COPPER BOILER.

Boiler to be made of Sheet Copper weighing 22 Oz. per Sq. Ft., Tinned Inside.
All Seams to be Lapped where possible. Hard Solder only to be used.



APPARATUS FOR MAKING ACCELERATED TEST.

To be made of Sheet Copper weighing 22 Oz. per Sq. Ft., Tinned Inside.
All Seams to be Lapped where possible. Hard Solder only to be used.

FIG. 8.

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.

JANUARY 18TH, 1911.

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NEW YORK SECTION SOCIETY FOR CHEMICAL INDUSTRY

Method Suggested for the Analysis of Limestones, Raw Mixtures and Portland Cements by the Committee on Uniformity in Technical Analysis with the Advice of W. F. Hillebrand.

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 fifteen 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 a 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 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 HF 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 HCl, 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 precipitated iron and aluminum hydrates, after settling, are washed once by decantation and slightly on the filter. Setting aside the filtrate,

* 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.

† For ordinary control in the plant laboratory this correction may, perhaps, be neglected; the double evaporation never.

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 of $KHSO_4$, 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 HF and H_2SO_4.† The filtrate is reduced by zinc, or preferably by hydrogen sulphide, boiling out the excess of the latter afterwards while passing CO_2 through the flask, and titrated with permanganate.‡ The strength of the permanganate solution should not be greater than .0040 grm. Fe_2O_3 per c.c.

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

* 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 HF 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.

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 blasted 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.

* 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.

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 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 Na_2CO_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 $\frac{3}{5}$ 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.

* Evaporation to dryness is unnecessary, unless gelatinous silica should have separated, and should never be performed on a bath heated by gas; vide Hillebrand.

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