EFFECT OF COMPOSITION ON QUALITY

Too high proportion of lime (lime factor of mix above 2.8) will give a slow-setting cement which will fail in the boiling test. If the excess of lime is great, pats of cement kept in cold water will show radial expansion cracks at the edges after a certain time, perhaps even within a few days. The same defects result from *imperfect grinding of the raw material*, and are far more often due to this cause than to excess of lime. Cement which is unsound and shows expansion from either cause may be improved and perhaps made sound by storage or by exposure to air. It is not, however, safe to rely greatly on this remedy. Lack of soundness is in all cases due to faulty manufacture, since well-burned cement made from suitably prepared raw material will invariably pass all soundness tests when fresh from the grinding mills. Consumers are advised to accept no cement which fails to pass a reasonable boiling test, as they will thus err, if at all, on the safe side, and will influence careless manufacturers to improve their process.

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

Overburning rarely occurs except with over-clayed mixtures or in consequence of the fluxing action of the fuel-ash or the brick lining of the kiln Properly proportioned mixtures stand a very high heat without injury.

Underburning, as stated above, in the case of an over-clayed mixture, yields quick-setting and weak cement. Normal mixtures, when underburned, usually give cement which fails in soundness tests. Light burning is generally indicated by heating of the cement on mixing with water. This behavior generally accompanies quick setting, and may be so marked as to be quite apparent to the touch of the fingers. Some cements, though slow-setting when first made, become very quick-setting on storage. Cases are on record in which this change has taken place within a few days. After longer periods the original slow-setting quality may return. The cause of this phenomenon has not been determined; it may be said, however, that troubles of this class, including quick setting and heating with water, are especially characteristic of cements made from aluminous materials.

CHAPTER VII

STANDARD CEMENT TESTS

The tests which are regarded as most suitable for the selection and acceptance of cement for important concrete construction are as follows:

Chemical analysis.
Specific gravity.
Fineness.
Activity, or time of setting.
Tensile strength of neat cement and sand mortars.
Soundness or constancy of volume.

The French Commission* in 1893, in addition to these tests, gave standard rules for testing weight, homogeneity (with the microscope), compressive strength, bending strength, yield of paste and mortar (*rendement*), porosity, permeability, decomposition, and adhesion, one or more of which tests may be desirable under certain conditions. As these are usually of minor importance, however, special mention of them is reserved for the following chapter.

In unimportant construction it is often safe to use a first-class American Portland cement without testing, and in other cases the test for soundness is the only one which need be actually made. Under almost all circumstances, however, when purchasing cement, full specifications (see Chapter III, p. 28) are advisable, so that if the cement does not work satisfactorily it may be more carefully examined and unused portions rejected.

In this chapter are presented, in addition to the description of the methods of making cement tests, complete lists of apparatus for a large and a small laboratory (p. 80), formulas and tables for determining the quantity of water in cement mortars (p. 85), comparisons of American and European practice in cement testing, a discussion of the causes of unsoundness and the results of soundness tests (p. 101), curves showing the growth in strength of typical cements and cement mortars (p. 99), and other information with reference to the qualities and testing of Portland cement.

STANDARD METHODS OF CEMENT TESTING

The following recommendations for testing are reprinted, with comments by the authors, from the preliminary or Progress Report of Special Com-

*Commission des Méthodes d'Essai des Matériaux de Construction, 1894, Vol. 1, p. 235.

mittee on Uniform Tests of Cement of the American Society of Civil Engineers,* as presented in 1903 and amended in 1904 and 1908. The methods are designed particularly for the testing of Portland cement, but are applicable to Natural (and also to Puzzolan), with the exception of paragraphs 5, 6, 68, (2), 71, and 74.

The standards which should be attained by first-class Portland and Natural cements are presented in the Standard Specifications in Chapter III, page 28.

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.

FIG. 8. Sampling Iron. (*See p. 64.*)

A sampling iron is illustrated in Fig. 8.

With the usual packing of Portland cement, four bags to the barrel, one bag in forty is equivalent to one barrel in ten. There is no necessity because of the smaller size of the packages for testing a larger proportion of the total weight.

The practice of mixing samples taken from a number of packages is by many authorities not considered advisable except for the purpose, suggested above, "of determining the characteristics of a shipment." A mixture of samples will not reveal irregularities in quality.

Chemical Analysis. 5. *Significance.* — Chemical analysis may render valuable service in the detection of adulteration of cement with considerable

*Proceedings, January, 1903.

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, Page 60, 1903, and The Engineering Record, Vol. 48, Page 49, 1903, is recommended.*

An exceedingly simple test for determining adulteration with raw or partially burned rock, is the treatment of the cement with muriatic acid as described in the purity test on page 4. It does not furnish the percentage of foreign ingredients, but the black precipitation of the adulterant darkens the color of the yellow jelly to a degree depending upon the quantity of adulteration.

Specific Gravity. 8. *Significance.* — The specific gravity of cement is lowered by underburning, adulteration and hydration, but the adulteration must be in considerable quantity to affect the results appreciably.

9. Inasmuch as the differences in specific gravity are usually very small, great care must be exercised in making the determination.

10. *Apparatus and Method.* — The determination of specific gravity is most conveniently made with Le Chatelier's apparatus. This consists of a flask (*D*), Fig. 9, 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.

*Printed in Appendix I of this Treatise.

12. The specific gravity can be determined in two ways:

(1) The flask is filled with either of these liquids to the lower mark (*E*), and 64 grams (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 to the top of the bulb (*C*)], until the upper mark (*F*) is reached.

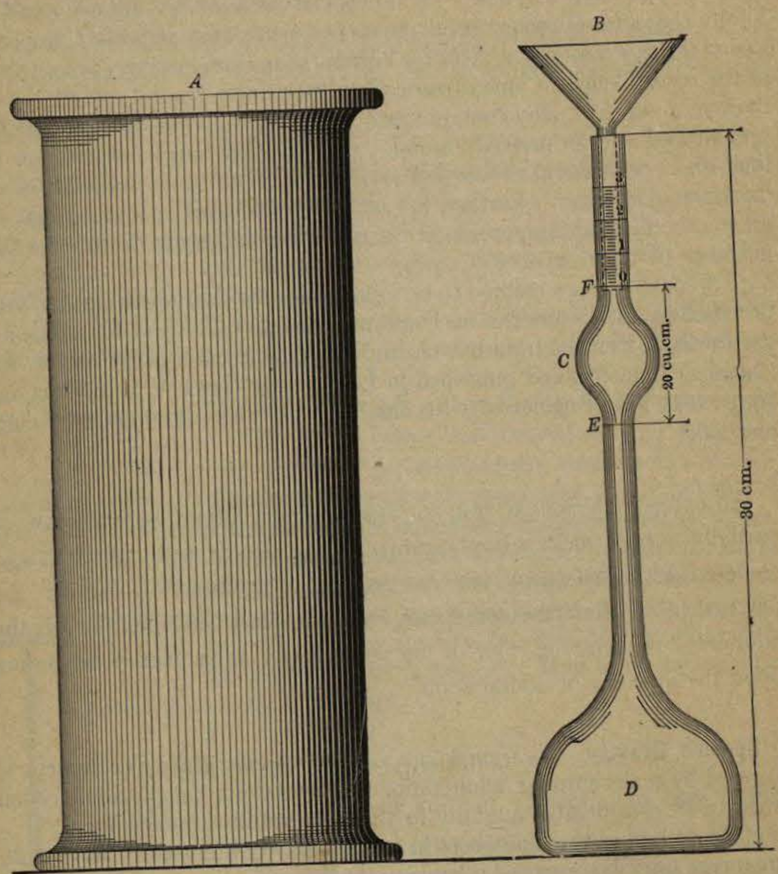

FIG. 9.—Le Chatelier's Specific Gravity Apparatus. (*See p.* 65.)

The difference in weight between the cement remaining and the original quantity (64 g.) is the weight which has displaced 20 cu. cm.

13. (2) The whole quantity of the powder is introduced, and the level of the liquid rises to some division of the graduated neck. This reading plus 20 cu. cm. is the volume displaced by 64 grams of the powder.

14. The specific gravity is then obtained from the formula:

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

15. 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.

16. 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 and repeating the operation.

17. More accurate determinations may be made with the picnometer.

The usual specific gravities of different classes of cement are given on page 81.

Le Chatelier's apparatus, suggested above as most convenient, was also recommended by Mr. E. Candlot after experiments for the French Commission,* in which he employed for comparison the Mann, Keate, Schumann, and Candlot, as well as the Le Chatelier apparatus.

Mr. Daniel D. Jackson† has more recently devised an apparatus with which temperature corrections can be made more readily than with the older types.

Fineness. 18. *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.

19. 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.

20. *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.

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

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

22. 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 " " " "

23. 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.

*Commission des Méthodes d'Essai des Matériaux de Construction, 1895, Vol. IV, p. 15.

†See *Engineering Record*, July 16, 1904, p. 82.

24. *Method.* — The Committee, after careful investigation, has reached the conclusion that mechanical sieving is not as practicable or efficient as hand work, and, therefore, recommends the following method:

25. 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% 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.

Laboratory scales for weighing the samples and the residue are illustrated in Fig. 10.

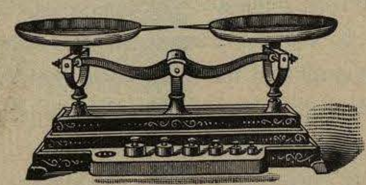

FIG. 10.—Delicate Laboratory Scales. (*See p.* 68.)

A table is given on page 84 for comparing American and European sieves, and the effect of the fineness of cement upon its strength is discussed on page 82.

It is impracticable to sift cement through a sieve finer than 200 meshes per linear inch. The particles which will just pass a No. 200 sieve are about 0.10 millimeter (0.004 in.) in diameter*. For still further separating the cement, some method based on the principle of suspension in liquid is employed as described on page 85.

Normal Consistency. 26. *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.

27. 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.

28. Various methods have been proposed for making this determination, none of which has been found entirely satisfactory. The Committee recommends the following:

*Allen Hazen in Report Massachusetts State Board of Health, 1892.

†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.

29. *Method. Vicat Needle Apparatus.* — This consists of a frame (*K*), Fig. 11, 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 grams (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.

30. In making the determination the same quantity of cement as will be subsequently used for each batch in making the briquettes (but not less than 500 grams) (17.64 oz.) are kneaded into a paste, as described in Paragraph 58, and quickly formed into a ball with the hands, completing the

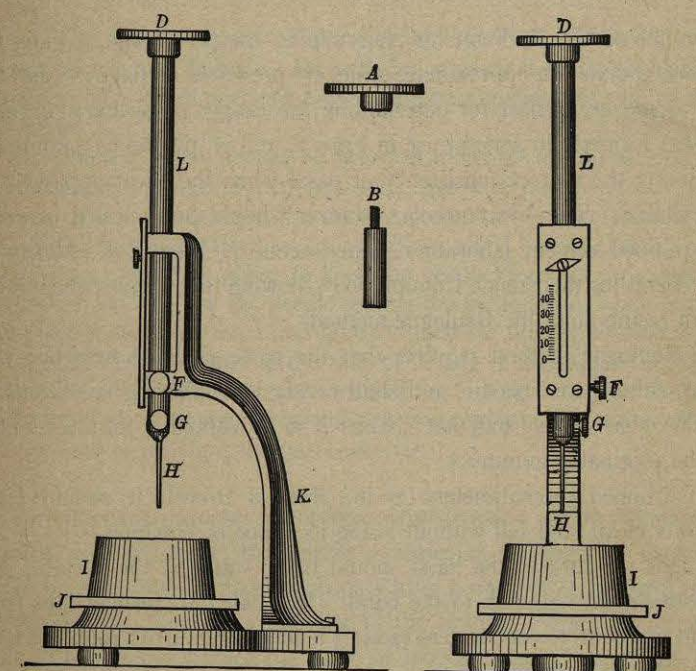

FIG. 11.—Vicat Needle. (*See p.* 69.)

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.

31. The paste is of normal consistency when the cylinder penetrates to a

point in the mass 10 mm. (0.39 in.) below the top of the ring. Great care must be taken to fill the ring exactly to the top.

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

33. 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 molding are likely to be less with such a paste.

34. Having determined in this manner the proper percentage of water required to produce a paste of normal consistency, the proper percentage required for the mortars is obtained from an empirical formula.

35. The Committee hopes to devise such a formula. The subject proves to be a very difficult one, and, although the Committee has given it much study, it is not yet prepared to make a definite recommendation.

Formulas of Mr. R. Feret for determining the percentage of water for sand mortars, and an approximate table, are presented on pages 86 and 88.

The Boulogne Method for determining the proper consistency of neat paste was formerly in general use in France, and is still the best guide for determining the correct consistency of paste when the Vicat apparatus is not available. The Vicat needle, however, should be included in every well equipped cement laboratory, experiments by Messrs. P. Alexandre and R. Feret for the French Commission* showing that it gives much more uniform results than the Boulogne method.

The Boulogne method requires that the paste shall be firm but well bonded, shining and plastic, and shall satisfy the following conditions:

1. The consistency shall not change if it is worked 3 minutes longer than the original 5 minutes.†

2. If dropped 50 centimeters (20 in.) from a trowel, it should leave the trowel clean, and fall without losing its shape or cracking.

3. Light pressure in the hand should bring water to the surface, and the paste should not stick to the hand. If a ball thus formed falls from a height of about 50 centimeters (20 in.) it should retain its rounded form without showing cracks.

4. The proportion of water should be such that more or less will produce opposite effects from those just described for the proper consistency.

Time of Setting. 36. *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

*Commission des Méthodes d'Essai des Matériaux de Construction, 1895, Vol. IV, p. 49.

†The original working for the U. S. Standard tests is $1\frac{1}{2}$ minutes (see paragraph 58).

"final" or "hard set"). The former of these is the more important, since, with the commencement of setting, the process of crystallization or hardening is said to begin. As a disturbance of this process may produce a loss of strength, it is desirable to complete the operation of mixing and molding or incorporating the mortar into the work before the cement begins to set.

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

38. *Method.* — For this purpose the Vicat Needle, which has already been described in Paragraph 30, should be used.

39. In making the test, a paste of normal consistency is molded and placed under the rod (*L*), Fig. 11, as described in Paragraph 31; this rod, bearing the cap (*D*) at one end and the needle (*H*), 1 mm. (0.039 in.) in diameter, at the other, weighing 300 g. (10.58 oz.). The needle is then carefully brought in contact with the surface of the paste and quickly released.

40. 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.

41. 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.

42. 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.

43. 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 molding the paste receives.

For practical purposes in ordinary construction where laboratory apparatus is unavailable, the setting qualities of a cement or mortar may often be examined by making up pats from a number of the packages and trying their hardening by pressure of the thumb. When the thumb nail fails to indent the surface the paste or mortar may be considered to have reached its final set.

The Gillmore needles, described on page 89 and there compared with the Vicat apparatus, were formerly the U. S. standard.

Standard Sand. 44. The Committee recognizes the grave objections to the standard quartz now generally used, especially on account of its high percentage of voids, the difficulty of compacting in the molds, and its lack of uniformity; it has spent much time in investigating the various natural sands which appeared to be available and suitable for use.

45. For the present, the Committee recommends the natural sand from

Ottawa, Ill., screened to pass a sieve having 20 meshes per linear inch and retained on a sieve having 30 meshes per linear inch; the wires to have diameters of 0.0165 and 0.0112 in., respectively, *i. e.*, half the width of the opening in each case. Sand having passed the No. 20 sieve shall be considered standard when not more than one per cent. passes a No. 30 sieve after one minute continuous sifting of a 500-gram sample.*

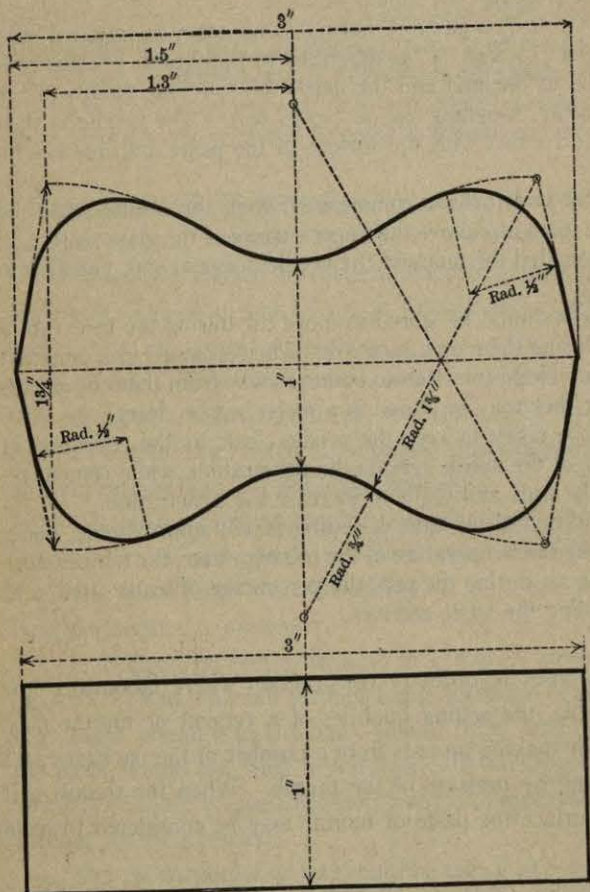

FIG. 12.—Details for Briquette. (*See p.* 72.)

Photographs of the grains of Ottawa and of crushed quartz sand are shown on page 175.

European is compared with U. S. standard sand on page 90.

Form of Briquette. 46. While the form of the briquette recommended by a former Committee of the Society is not wholly satisfactory, this Committee is not prepared to suggest any change, other than rounding off the corners by curves of ½-in. radius, Fig. 12.

The German standard briquette is sketched on page 92.

Molds. 47. 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.

48. Gang molds, which permit molding a number of briquettes at one

*The Sandusky Portland Cement Company, of Sandusky, Ohio, has agreed to undertake the preparation of this sand, and to furnish it at a price sufficient only to cover the actual cost of preparation.

time, are preferred by many to single molds; since the greater quantity of mortar that can be mixed tends to produce greater uniformity in the results. The type shown in Fig. 13 is recommended.

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

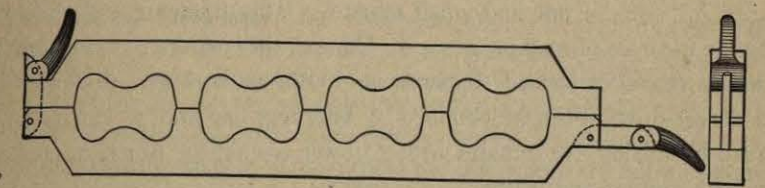

FIG. 13.—Details for Gang Mold. (*See p.* 73.)

Mixing. 50. All proportions should be stated by weight; the quantity of water to be used should be stated as a percentage of the dry material.

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

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

53. 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.

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

55. The Committee, after investigation of the various mechanical mixing machines, has decided not to recommend any machine that has thus far been devised, for the following reasons:

(1) The tendency of most cement is to "ball up" in the machine, thereby preventing the working of it into a homogeneous paste; (2) there are no means of ascertaining when the mixing is complete without stopping the machine, and (3) the difficulty of keeping the machine clean.

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

The apparatus required for mixing briquettes consists of a piece of 1-inch plate glass at least 24 inches square, counter scales (preferably metric system), recording from 10 grams to 1½ kilograms, a 250