The neat and 1 : 3 (*i. e.*, one part cement to 3 parts sand by weight) curves are averaged from over 100,000 briquettes, while the other curves are each based on tests of 300 to 500 briquettes.

The cements included a number of brands, American brands largely predominating. The sand was crushed quartz, the former U. S. standard. The Philadelphia records include tests of much longer time than one year, and there is a noticeable falling off in the observed tensile strength after the one-year period. This is most noticeable with neat cement of rotary kiln brands, but also occurs to a less degree with sand mortars. With cements from stationary kilns it is less marked. The falling off in tensile tests is generally attributed to the brittleness of the small sized specimens, which tends to irregularity of results with the ordinary testing machine, and to the unequal hardening of the surface and interior of the specimen, rather than to actual deterioration in the cement.

The average growth in strength of neat Natural cement and Natural cement mortars is illustrated in Fig. 29 from data kindly prepared by

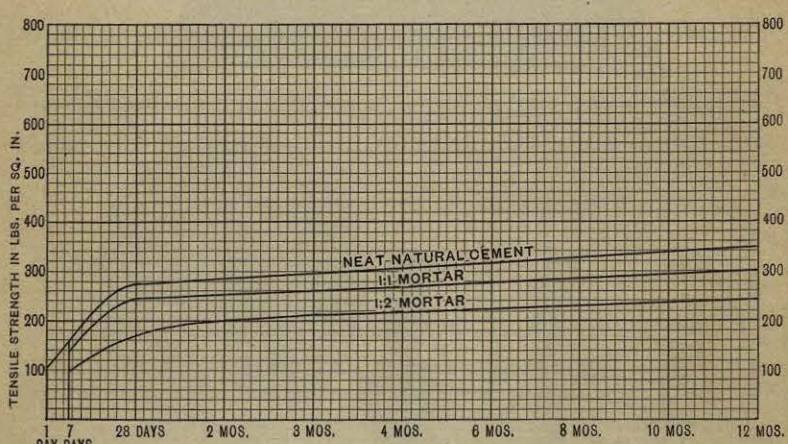


FIG. 29.—Growth in Tensile Strength of Neat Natural Cement and Natural Cement Mortars with Different Proportions of Standard Sand. (*See p.* 100.)
(From data by Richard L. Humphrey and A. W. Munsell.)

Mr. Richard L. Humphrey from Philadelphia tests, and by Mr. A. W. Munsell from tests made for the Baltimore & Ohio R. R. Cements from seven different sections of the United States are included in the averages from which the curves are drawn, representing the Akron, Cumberland, James River, Lehigh Valley, Louisville, Milwaukee, Rosendale and Utica districts.

SOUNDNESS OR CONSTANCY OF VOLUME

The term "soundness" is more commonly used in America and England than the expression "constancy of volume" suggested by the Committee of the American Society of Civil Engineers, or "deformation" as employed in France. The purpose of the test is to determine in advance whether a cement is in danger of disintegrating, that is, crumbling, or of expanding or contracting so as to cause distortion or cracking in the masonry.

If a cement satisfactorily passes the tests for soundness, it will in all probability withstand the effect of the elements without swelling or disintegration, and will continue to harden for an indefinite period. Failure, on the other hand, to pass the tests for soundness, especially the hot test, is not positive proof of inferiority, for a cement which fails to pass may possibly, through subsequent exposure to the air before being used, or because of mixing with sand or other aggregate, produce durable masonry. We may, however, with safety adopt the following conclusion:

If a Portland cement passes the hot test it may be used immediately with reasonable certainty of its ultimate soundness. If it fails to pass, it should be regarded with suspicion and thoroughly tested.

Causes of Unsoundness. Disintegration, or crumbling, of work in Portland cement properly mixed and laid, is usually due to an excess of lime in a form which can be attacked by the elements. This may come about in two entirely distinct ways, either (1) by the use of too high a proportion of lime in the raw materials from which the cement is made, (2) by under-burning the cement, or (3) by too coarse grinding.

The presence of magnesia in excess in a thoroughly burned cement may produce a gradual expansion which will disintegrate the mortar or concrete after several years. This action, brought to notice by tests of Mr. H. Le Chatelier,* is generally recognized, but opinions differ as to the limit to the percentage of magnesia which may occur in Portland cement without deleterious effect. Le Chatelier's experiments led him to consider 5% as injurious. The Association of German Cement Manufacturers first placed the limit at 3½%, and later raised it to 5%. Mr. Spencer B. Newberry states (page 56) that recent experiments made by himself and by Van Blaese show that cements containing 8% or 9% of magnesia will pass the boiling test, while those with 15% magnesia will expand. The limit of 4% recommended by the Committee of the American Society for Testing Materials in 1904 (see p. 30) is undoubtedly conservative. Natural cement, which is burned at a lower temperature,

*Commission des Méthodes d'Essai des Matériaux de Construction, 1895, Vol. IV, p. 229.

may contain a much larger quantity of free lime and of magnesia without injury.

The expansion caused by an excess of free lime is due to the hydration or slaking of the calcium oxide (CaO). This is readily understood from the expansion of common lime, which in slaking with water will produce a bulk of paste from 2 to 3 times greater than the volume of the loose powder. The presence of lime in a free or loosely combined state must not be confounded with other compounds of calcium. A thoroughly slaked lime paste, or powder, that is, one which is completely hydrated, may in fact be added to a Portland cement mortar without injurious results, to lengthen its time of setting or to produce a more water-tight mixture.

The small amount of free lime which frequently occurs and sometimes produces unsoundness in first-class Portland cement, tested when fresh, may be hydrated and rendered harmless by air-slaking after, say, two or three weeks' storage, or after spreading the cement out in the air.

Adulteration with slag may cause a cement containing an excess of free lime to pass the boiling test.

Tests for Soundness. The presence of ingredients which will render a cement unsound, that is, which will cause it to expand or disintegrate, is determined by the eye, or by measuring appliances in specimens which have been exposed under conditions which as nearly as possible produce the same effect as the practical effects of time and the elements.

There is apparently no reliable method for determining the presence of free lime by chemical analysis. Mr. E. Candlot* says that "there is in fact no method for finding the percentage of free lime in the cement," and Dr. Schuman* concurs in this view in the following statement·

> I do not know a method for finding out the percentage of free lime in Portland cement. I do not think there exists such a method, and I am myself of the opinion that chemists will never find out one; the solutions capable of taking away the free lime from the cement will always work in a more or less strong degree on the cement itself.

This inability to detect free lime by chemical analysis necessitates a resort to physical tests. Specimens for testing soundness are generally circular pats tapering toward the edges, so that the expansion of the mass will tend to enlarge the circumference and thus produce cracks at the edges.

*Quoted by W. W. Maclay in Transactions American Society of Civil Engineers, Vol. XXVII, p. 448.

Egg-shaped specimens and also briquettes are sometimes used. Both of these show deterioration by the appearance of the surface.

Appearance of Soundness Specimens. Cracks which appear on pats are not always caused by unsoundness. Expansion cracks, which reveal an unsound cement, are distinguished from shrinkage cracks, which usually appear during setting instead of after the cement is set, in Figs. 30 to 37. Hair cracks also sometimes appear upon specimens, and in practice upon neat cement or very rich mortar, where so large an excess of water has been employed in mixing that it does not dry off until the cement has set, and causes the deposition of a very thin coating of partially decomposed cement which had remained in suspension in the water. An unsound cement in air or in water at the ordinary temperature will generally show defect within 28 days, although in very exceptional cases several months or even years have been known to elapse before signs of deterioration appear in specimens which have not been subjected to heat.

Photographs of pats illustrating the appearance of defective specimens which have been subject to the boiling test are shown in Figs. 38 and 39, pages 108 and 109. Figs. 30 to 37, pages 104 and 105, are sketches* employed in the Philadelphia Municipal Laboratories for distinguishing harmless appearances in neat pats from evidences of unsoundness. Mr. Taylor describes the pats as follows:

> Fig. 30 represents a normal pat in good condition.
>
> Fig. 31 represents shrinkage cracks. These cracks are ordinarily due to the use of a too wet mixture or to too quick a drying out. If the pats are left exposed to dry air during setting these cracks are often developed. Shrinkage cracks ordinarily, therefore, indicate only a lack of care in manipulation, and not dangerous properties in the cement.
>
> Fig. 32 shows cracks caused by the expansion of the cement and the curling of the edges of the pat from the glass while the pat still adheres, which is often coincident with the expansion. In the air pats these cracks are developed in nine-tenths of the pats adhering to the glass, and unless very decidedly marked are not dangerous. They should not exist in the water pats. If they do exist, however, to an appreciable extent, it denotes the presence of a too great proportion of expansives, which ordinarily is sufficient to condemn the sample.
>
> Fig. 33 shows blotching, a pat which is usually indicative of either adulteration or under-burning. This condition in itself should not necessarily mean rejection, but should always induce an investigation of the causes producing it, which may or may not be sufficient to warrant rejection.
>
> Fig. 34 shows pats which have left the glass (*A*) by mere lack of adhesion, (*B*) by contraction, and (*C*) by expansion. (*A*) is never dangerous

*Presented to the authors by Mr. W. Purves Taylor.

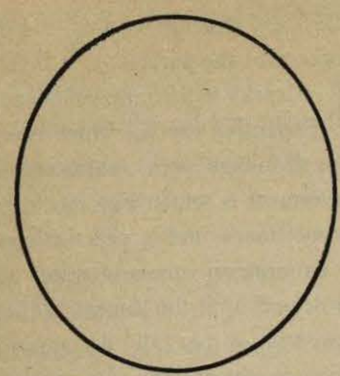


FIG. 30. Normal Pat.*

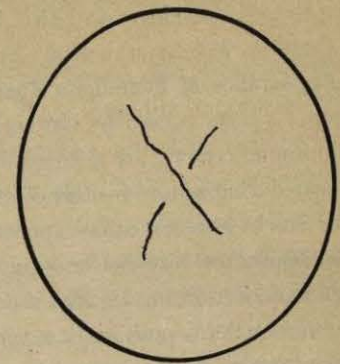


FIG. 31.—Harmless Shrinkage Cracks *

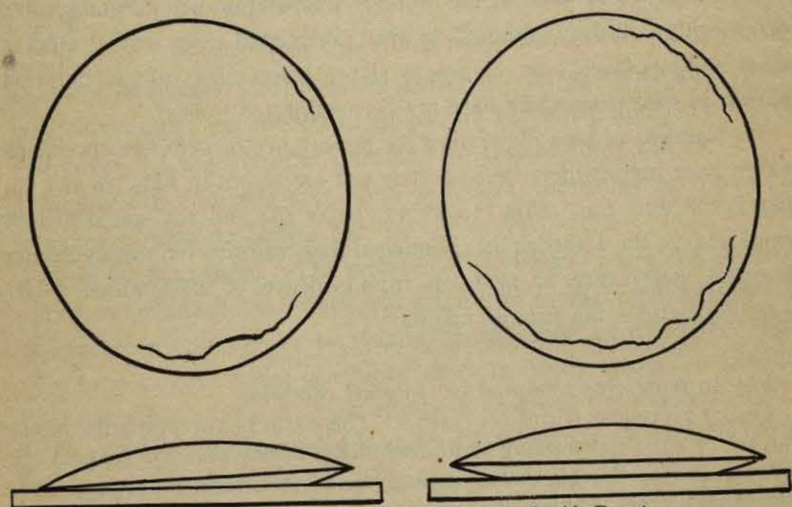


FIG. 32.—Expansion Cracks, Harmless in Air Pats.*

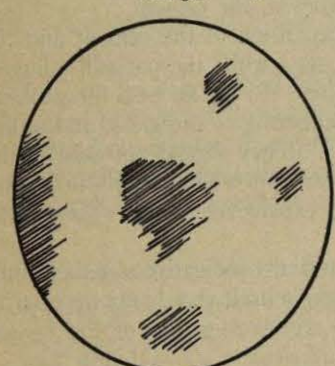


FIG. 33.—Blotches Requiring Investigation.*

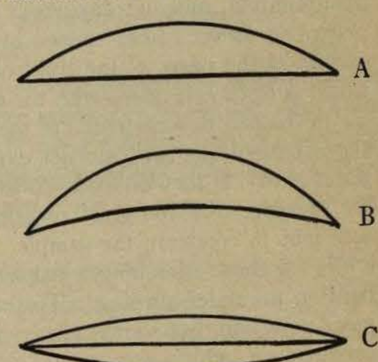


FIG. 34.—Pats which have left Glass.*

*See pp. 103 and 106.

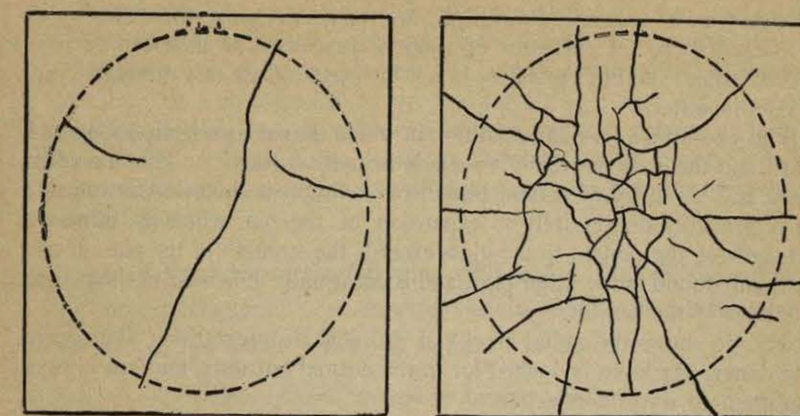


FIG. 35. —Cracked Glass (pat removed.)*

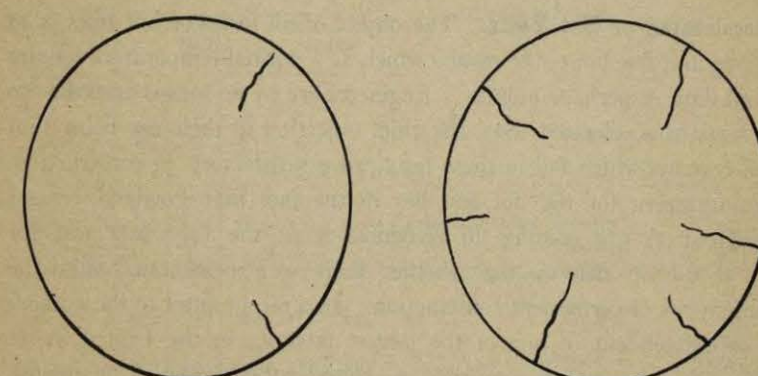


FIG. 36.—Incipient Disintegration.*

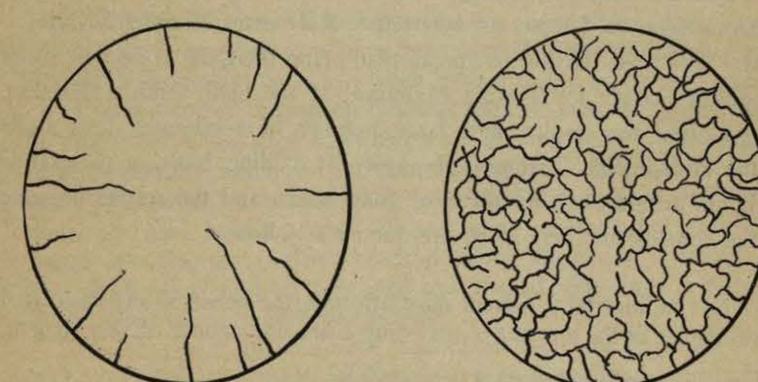


FIG. 37.—Complete Disintegration.*

*See pp. 103 and 106.

in either air or water. (*B*) and (*C*) are dangerous only when existing in a marked form. A curvature of about a quarter of an inch can be considered about the limit of safety in a 3-inch pat. Case (*C*) rarely, if ever, occurs in water.

Fig. 35 shows a peculiar condition in which the pat is perfectly sound and hard, but the glass on which it is made is badly cracked.* This has often been laid to chemical action, but this conclusion is doubtless erroneous. It is probably due entirely to expansion of the pat, when the adhesive strength of the cement to the glass exceeds the strength of the glass itself. It is only found in the water pats, and is not usually indicative of dangerous qualities of the cement.

Fig. 36 shows the radial cracks of incipient disintegration. These are the danger marks to be looked for in the normal pat tests, and are always sufficient to warrant rejection.

Fig. 37 shows cases of complete disintegration, which almost invariably begins merely by showing radial cracks, as in Fig. 36.

Accelerated or Hot Tests. The object of all forms of hot tests is to produce in a few hours the results which at a normal temperature require several days or perhaps months. Engineers are by no means agreed as to the value of accelerated tests, the chief objection to their use being that some cements which fail in these tests prove satisfactory in construction.

An argument for the hot test lies in the fact that Portland cement manufacturers are coming to recognize it as the very best test for them to use in determining whether their own cement will fulfil the requirements of permanent construction. In a recent letter to the authors the superintendent of one of the largest factories in the United States writes, "So far as we are concerned, we consider the hot test of the greatest importance. If this shows up well, we feel quite satisfied that all other tests will show up properly." Those desiring to investigate the various opinions upon the subject are referred to References, Chapter XXXI.

Mr. W. Purves Taylor, in a paper read before the Cement Section of the American Society for Testing Materials, at the Sixth Annual Meeting, 1903,† gives the results of a large number of accelerated tests made at the Philadelphia Testing Laboratory by boiling balls or pats (after 24 hours in moist air) for three or four hours, and the results of some of the conclusions there given are quoted as follows:

"The condition in a cement most affecting the result of an accelerated test is its age or the amount of seasoning it has undergone. Every cement,

*Similar causes may produce one or two cracks in the glass.

†Proceedings American Society for Testing Materials, 1903, Vol. III, p. 374, also printed in *Engineering News*, July 23, 1903, p. 81.

no matter how well proportioned and burned, will contain at least a small amount of free or loosely combined lime, which will usually cause unsoundness if used or tested at once. This lime, however, will hydrate in a very short time on exposure to air, thus rendering it inert and preventing any expansive action. It will, therefore, be found in a large majority of cases that if a cement failing in the accelerated tests be stored for two or three weeks, this unsoundness will disappear, and the cement pass the test with ease."

This is illustrated in the following table and in Fig. 38, page 108, the first three photographs also showing various conditions which may be expected in specimens which fail to pass accelerated tests.

Effect of Age of Cement on Results of Boiling Test.

BY W. PURVES TAYLOR. (*See p. 107.*)

Age of cement when tested	Tensile Strength					Normal Pat Tests		Boiling Test
	Neat			1:3 sand				
	1 day	7 days	28 days	7 days	28 days	28 days in air	28 days in water	
1 week	550	765	762	171	225	Curled and soft.	Slightly checked.	Partly disintegrated.
2 weeks	548	67	771	17.	246	Slightly curled.	Slightly curled.	Checked and cracked.
3 "	492	718	763	182	244	"O. K."	"O. K."	Slightly checked.
5 "	427	692	747	183	249	"O. K."	"O. K."	Sound.

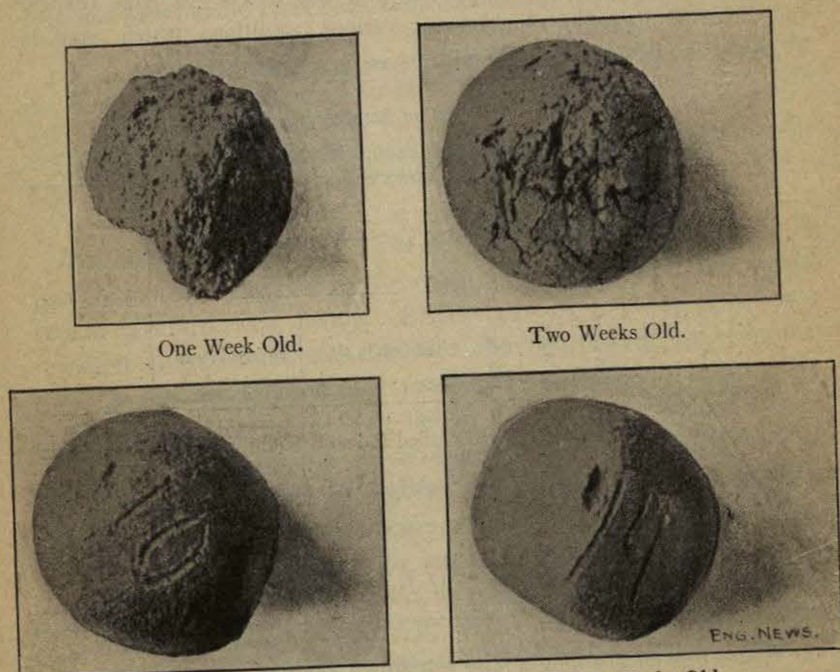
"Coarseness of grinding is also a frequent cause of unsoundness for the reason that the larger particles are not readily susceptible to hydration, and contain for a long period of time expansive elements which very rapidly develop a disintegrating action when treated in the accelerated tests."

"A large number of tests on different cements were made and the time at which failure occurred was observed. In these tests it was found that of those samples which did not pass the test, 22% failed in the first half hour, 57% failed in the first hour, 85% failed in two hours, 96% in three hours, and 99% in four hours," "thus showing generally that a test piece of cement standing three or four hours of boiling will almost invariably stand a much greater length of time, and also that at least three or four hours should always be allowed for the test."

"Pats of cement allowed more than about twelve hours to harden will, if unsound, fail when tested by boiling at almost any time in the future."

"We now come to the very important question of the relation of the boiling tests to the other tests for soundness and strength as made in the laboratory. No one who has had much experience with the boiling test questions that, although it is by no means infallible, the results obtained from it are generally corroborated by either the tensile tests or the normal tests for soundness. The writer has recently compiled some data in regard to this point, covering over a thousand tests on many varieties of cement, with the following results:

"Of all samples failing to pass the boiling test, 34% of them developed checking or curvature in the normal pats — or a loss of strength in less than twenty-eight days. Of those samples that failed in the boiling test but re-



One Week Old. Two Weeks Old.

Three Weeks Old. Five Weeks Old.

FIG. 38.—Specimens showing the Effect of the Age of the Cement upon its Soundness. (*See p.* 107.)

mained sound at twenty-eight days, 3% of the normal pats showed checking or abnormal curvature in two months, 7% in three months, 10% in four months, 26% in six months, and 48% in one year; and of these same samples, 37% showed a falling off in tensile strength in two months, 39% in three months, 52% in four months, 63% in six months, and 71% in one year. Or, taking all these together, of all the samples that failed in the boiling test, 86% of them gave evidence in less than a year's time of possessing some injurious quality.

"On the other hand, of those cements passing the boiling test, but one-half of 1% gave signs of failure in the normal pat tests, and but 13% showed a falling off in strength in a year's time.

"This certainly makes a very strong showing in favor of the boiling test, at least considered from a laboratory standpoint.

"In order to show the great value sometimes obtained from the results of the boiling test, several examples are given in the table on page 110 of tests of cements occurring in the regular routine work of the laboratory."

The air and water pats of sample 2 of this table are shown in Fig. 39 at the age of four months. These pats were sound at twenty-eight days.

In conclusion Mr. Taylor lays special emphasis upon the fact that many cements which do not pass the boiling test will give excellent results in

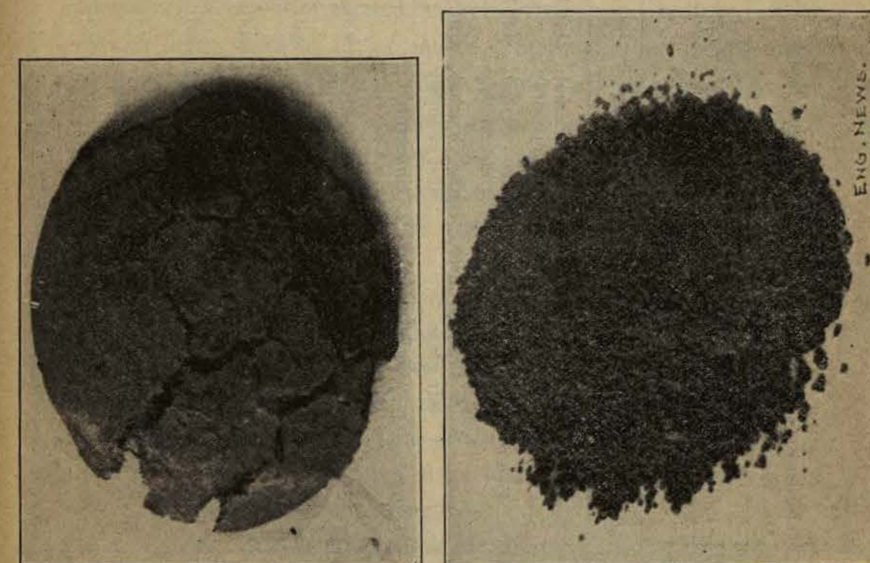


FIG. 39.—Examples of Unsound Pats at 4 months which were sound at 28 days. (*See p.* 109.)

practice. He gives as the probable reason for this that the test for soundness is generally made immediately upon the receipt of a shipment, while the cement used in construction has opportunity to season, and upon the fact "that the disintegrating action of a cement is always far greater when mixed neat than when mixed with an aggregate, and the greater the amount of the aggregate the less the tendency to unsoundness." It is often good policy before rejecting a cement which fails to pass the hot test to hold it for a week or two so that it may further season and then retest it.

Methods of Making Accelerated Tests. The methods of conducting accelerated tests are numerous, the object of all of them being to hasten

Evidences of Failure in Cement Indicated by the Boiling Test.
By W. Purves Taylor. (*See p.* 109.)

TENSILE STRENGTH							NORMAL PAT TESTS				
Neat				1:3 sand			Air		Water		Boiling test
1 day	7 days	28 days	4 months	7 days	28 days	4 months	28 days	4 months	28 days	4 months	
522	793	797	Disintegrated.	204	257	52	Very slightly curled; left glass.	Badly curled; soft and crumbly.	Left glass.	Disintegrated.	Disintegrated.
503	872	586	Disintegrated.	184	239	47	Very slightly curled; left glass.	Badly curled; soft and crumbly.	Left glass.	Disintegrated.	Disintegrated.
498	762	700	Disintegrated.	176	231	119	Very slightly curled; left glass.	Badly curled; soft and crumbly.	"O. K."	Disintegrated.	Disintegrated.
427	751	603	223	183	227	94	Very slightly curled; left glass.	Disintegrated.	"O. K."	Disintegrated.	Disintegrated.
503	827	717	177	220	252	132	Left glass.	Badly curled; soft and crumbly.	"O. K."	Disintegrated.	Disintegrated.
492	883	620	202	195	217	147	Left glass.	Badly curled; soft and crumbly.	"O. K."	Disintegrated.	Badly checked.
535	864	743	94	197	241	77	Very slightly curled; left glass.	Badly curled; soft and crumbly.	"O. K."	Badly curled and checked.	Checked and cracked.
502	829	722	320	203	247	65	Very slightly curled; left glass.	Badly curled; soft and crumbly.	"O. K."	Badly curled and checked	Checked and cracked.
Neat tests not made.				172	219	93	Very slightly curled; left glass.	Badly curled; soft and crumbly.	"O. K."	Badly curled and checked.	Checked.
Neat tests not made.				198	231	101	Left glass.	Badly curled; soft and crumbly.	"O. K."	Badly curled and checked.	Checked.

Note.—All of these cements were normal in specific gravity, time of setting, and fineness.

the hardening of the cement so as to produce in a few hours results which under ordinary conditions require weeks or months. Boiling the specimens, instead of steaming them as recommended by the Committee of the American Society of Civil Engineers, while more common, is more severe. Other methods are employed in Europe.

The Steam Test, recommended by the Committee of the American Society of Civil Engineers, requires, as already described (p. 77), that the pat after twenty-four hours in moist air shall be placed in an atmosphere of steam above boiling water.

The Boiling Test was originated by Prof. Tetmajer in Germany. After twenty-four hours in moist air, or until it is thoroughly set, the specimen is placed in cold water, which is raised to and then maintained at the boiling point for several hours. Three or four hours is the time specified by Mr. W. Purves Taylor, and often used in the United States, although some cement factories boil for twenty-four hours. Dr. Michaelis advocates six hours' boiling, and this period is specified by the French Commission.

Combined Boiling and Tensile Test. A regular test at many Portland cement factories consists in testing the tensile strength of briquettes which have been subjected to the hot test. A briquette of neat cement after twenty-four hours under a damp cloth is placed in an atmosphere of steam over boiling water for an hour or two, and then immersed in water at about the boiling point and boiled for about twenty-four hours, when it must show a certain tensile strength.

The Hot Water Test, as adopted by Mr. Henry Faija in England, and advocated there by Mr. David B. Butler, consists in subjecting a newly mixed pat to a moist heat of 100° to 105° Fahr. (38° to 40° Cent.) for six or seven hours, or until thoroughly set, and then placing it in warm water at a temperature of 115° to 120° Fahr. (46° to 49° Cent.) for the remainder of the twenty-four hours. Mr. Deval in France employed a temperature of 176° Fahr. (80° Cent.) for a period of six days.

Other Accelerated Tests which have been employed in Europe are oven tests, where the specimen is heated in an oven; glow tests, where a ball is heated over a gas flame, and Prussing disc tests, where discs are formed under heavy pressure and then exposed to hot water.

Measurement of Expansion. Appliances have been devised for testing the soundness of cement by measuring the amount of expansion or deformation which it undergoes in different periods of time. The principal of these are the long bar apparatus, devised by Messrs. Durand-Claye and

Debray, which was recommended by the French Commission, Bauschinger's caliper apparatus, and Le Chatelier's tongs.*

The Chimney Expansion Test, in which a small quantity of neat cement is solidly pressed into a straight lamp chimney with the idea that an unsound cement will break the glass, is worthless, as all first-class cements expand to a greater or less degree.

*Described in Spalding's Hydraulic Cement, 1903, p. 166.

CHAPTER VIII

SPECIAL TESTS OF CEMENT AND MORTAR

The most important tests for comparing the qualities of different cements and for determining their practical value have been described in the preceding chapter. Certain other tests are often made to investigate special qualities of a cement or mortar, or for scientific research.

Such special tests may be enumerated as follows:

- Color.
- Weight.
- Microscopical.
- Compressive.
- Transverse.
- Adhesive.
- Shearing.
- Abrasive.
- Porosity.
- Permeability.
- Yield of mortar.
- Rise in temperature.

COLOR

The color of a cement bears but slight relation to its quality, but a variation of color in the same brand is sometimes an indication of inferiority. Natural cements made in different localities may often be distinguished from each other and from Portland cements by their color.

Portland Cement. The chemical composition of Portland cements made by different processes is so uniform that the color of different brands varies less than that of Natural cements.

The color of Portland cement is described as a cold blue gray. In England the term "foxy" is applied to a Portland cement of a brownish color. According to Mr. David B. Butler* this denotes "insufficient calcination or the use of unsuitable clay or possibly excess of clay." He further states that if a Portland cement contains a large quantity of underburned particles, on account of their lower specific gravity they tend to rise to the surface on troweling, thus forming a yellowish brown film which is noticeable in the section of the briquette after fracture.

*Butler's Portland Cement, 1899, p. 255.