

MANUAL OF CEMENT TESTING¹

INTRODUCTION

This Manual of Cement Testing is intended to supplement—not in any way to supersede—the various ASTM standard methods of sampling and testing of hydraulic cement. This manual was prepared by Committee C01 on Cement and has been accepted by the Society for publication as information only. The manual is not a part of the ASTM methods.

The ASTM standard methods of testing cement are essential adjuncts to the specifications, because poorly written test methods may lead to erroneous conclusions as to the quality of the cement.

Precautionary statements are found in many of the test methods. Before using a test method for cement testing, the precautionary statement should be noted and clearly understood. **See Section 56 for safety precautions.**

The approved test methods are frequently revised, sometimes as often as twice a year. The cement tester must be sure the latest revision of the test method is used. Due to the frequency of change, often the most up to date standard may be found only through the ASTM website.

All methods of test have some variability and it must not be expected that exact agreement will be secured in tests by different operators, or by the same operator at different times, even when the tests are made on the same sample and under the same conditions. Many steps are involved in making cement tests, and the results are sometimes greatly influenced by seemingly minor variations in procedure.

It is the purpose of this manual to emphasize those factors which may affect results of tests, and to call attention to less apparent influences which are important but which are sometimes overlooked.

The ASTM standards for cement are intended to set forth minimum requirements that must be met. Cement testing laboratories should have on hand copies of current ASTM standards for cements, which include the standard methods of sampling and testing cement. The cement testers should be thoroughly familiar with these standards, and should use every care to follow the standards strictly and maintain their testing equipment in good condition. If careless methods and non-conforming equipment are used, the results may lead to the rejection of a good product or the acceptance of a product that may not conform to the specification requirements.

It is important to create an interest in standard methods and find effective ways to eliminating erroneous practices and call attention to defective apparatus. A suggested course of training for cement testers is given in Section 55.

After training, evaluation of the cement tester is important. The procedures and equipment of the cement test methods used by the cement tester should be evaluated. Evaluation programs are offered by the Cement and Concrete Reference Laboratory and by individual cement producers. Information on the inspection and proficiency sample programs are available in Section 54.

¹ This manual is under the jurisdiction of ASTM Committee C01 on Cement and is the direct responsibility of Subcommittee C01.97 on Manual for Cement Testing. Published as information, June, 1932; revised, 1944, 1946, 1950, 1952, 1954, 1955, 1956, 1958, 1960, 1968, 1986, 1989, 1990 and 1992; editorially corrected 1970, 1971, 1972, 1975, 1977, 1978, 1981, 1986, 2000, 2002, 2008, 2009, 2010, and 2011.

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SAMPLING

(See Practice C 183)^{1,2}

1. Treatment of Samples

1.1 Test samples must be forwarded to the laboratory promptly in airtight, moistureproof containers. Ordinary paper or cloth sacks must not be used. Poly(vinyl chloride) sample containers, upon occasion, have been found to affect the air entraining potential of a cement sample. The same problem might be experienced with containers made from other plastics. These containers should be used with caution. Each sample should be marked plainly and identified completely on the outside of the body of the container as to source and date of sampling. Do not mark the lids of containers, since the lids may be readily interchanged and lead to confusion. Also, a complete identification should be enclosed in the container. As it is important to keep variable influences at a minimum, the sample should be kept in airtight, moisture-proof containers from the time it is taken until it is tested or made into specimens. Reserve portions of samples should be likewise protected as long as they are kept.

1.2 The section on Amount of Testing of Practice C 183 prescribes the number of tests per lot for most properties. After a quality history has been established, testing may be carried out at the reduced rate as long as no pairs of results produce an average in the critical range. The critical range is so defined that when the average of a pair of tests is outside the range, there is a 95% probability that if a large number of samples were tested, fewer than 5% would fail the specification limit. It is based on the average range of results of pairs of samples accumulated during the development of the quality history. Where the specification limit is a minimum value, the critical limit is placed above the specification limit by an amount equal to the average range multiplied by 2.49. The critical range

includes all possible test results with values below the critical limit. Where the specification limit is a maximum value, the critical limit is below the specification limit, and the critical range includes all results above the critical limit. The critical limit is intended to ensure that the mean of the population of test results is removed from the specification limit by at least 1.645 standard deviations, that being the value corresponding to a failure rate of 5 %. Since the standard deviation of a sampling distribution of averages of pairs is equal to the population standard deviation divided by the square root of 2, the critical limit, which is the 95% confidence limit, must be removed from the desired position mean by a distance equal to $1.645/\sqrt{2}$ or 1.163 standard deviation. Thus, the distance between the critical limit and specification limit must be the sum of 1.645 and 1.163, or 2.808 standard deviations. Since, for a sample size of 2 the standard deviation is exactly equal to 0.886 range, the distance between the critical limit and the specification may be expressed as 2.49 average ranges.

1.3 Chapter 8 of The American Concrete Institute document, Guide to Selection and Use of Hydraulic Cements, ACI 225.1 R,³ is a review of sampling and testing procedures of hydraulic cement that are commonly found in the industry. The various aspects of sampling, testing, and reporting of data are discussed and may provide useful information on the several quality control and acceptance procedures that are commonly used.⁴

2. Preparation of Samples for Test

2.1 Prior to testing, a sample shall be thoroughly mixed and passed through the 850- μ m (No. 20) sieve or any other sieve having approximately 20 openings per inch, and brought to room temperature. All foreign matter and lumps that do not breakup on sieving and brushing easily must be discarded.

³ American Concrete Institute, P.O. Box 9094, Farmington Hills, MI 48333-9094. Available from American Concrete Institute (ACI), P.O. Box 9094, Farmington Hills, MI 48333-9094, <http://www.concrete.org>.

⁴ For additional information see the paper by Dywer, J. R., and Young, R. N., "Inspection of Portland Cement," *Concrete*, Vol 21, August and September 1992, pp. 56 and 95. This paper has been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR: RR:C01-1002.

² For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

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2.2 Note any unusual conditions of the sample.

FINENESS BY THE 150- μ m (No. 100) AND THE 75- μ m (No. 200) SIEVES

(See Test Method C 184)²

3. Accuracy of Sieve Tests

3.1 The requirement for 150 μ m (No. 100) and 75 μ m (No. 200) sieves has been deleted from the ASTM specification for portland cement. These fineness tests may be useful for research or special purposes, such as raw material preparation.

3.2 The operator must possess considerable skill, acquired only by practice in order to make satisfactory fineness determinations. With proper care, an experienced operator should check himself within 1 %, and generally within 0.5 %, on portions of the same thoroughly mixed sample on the same sieve in duplicate tests. If an accuracy of 1 % or better is required, standard calibrated sieves shall be used.

3.3 Remove the super fines from the sample before beginning the sieve test itself. This is most easily done by suspending the sieve between the fingers of each hand and shaking it loosely back and forth, alternately striking the palms of the hands. Five to ten minutes of sifting will clean the sample very adequately. Before beginning the 1-min shaking tests, it is important to see that no flour has blinded any of the screen openings. When performing the 1-min shaking test, each operator should experiment to determine the angle from the horizontal at which the sieve is to be held. When the proper angle has been reached, the residue distributes itself across the screen during each stroke so that most effective screening action is obtained. It is also important that the sieve is moved against the hand held in the upright position. Some operators take liberties with the test and strike the sieve with the palm of the open hand. Screening is not complete when this is done. The weight of residue will tend to be high. The rate at which the sieve strikes the palm is important. Take a full 10 s for each 25 strokes in each of the six positions. Do not try to do the 125 strokes and the five rotations in 1 min. When this is done the stroke rate is speeded up and there is a tendency to strike the palm less hard, which again tends toward high residues.

4. Design of Sieves

4.1 It is essential that all sieves used in the tests of fine materials be well designed and constructed and that they be provided with closely fitting covers and pans in order to avoid the loss of small quantities of material during the sieving operation.

5. Care of Sieves

5.1 Sieves should be examined frequently for defects in the cloth. Holes or breaks are sometimes indicated by very noticeable irregularities in the end-point test weighings. Holes are sometimes found in the soldered joint between the sieve and frame. Soldering must be done with great care so that the wire cloth is not injured by excessive heat or by harmful fluxes. Sieves should be kept thoroughly clean and dry. The battering of edges of sieve frames and pans should be avoided, for this will interfere with the proper fitting together of pan, sieve, and

cover. The brush used for cleaning the sieve should be stiff enough to clean cloth, but not so heavy as to damage it. Be careful that the brush is not so held that the metal binding will drag across the sieve cloth and press objectionable ridges in it.

5.2 New sieves should be cleaned with a suitable solvent or detergent to remove any grease before making tests. Alcohol may attack the lacquer on some sieve frames.

FINENESS BY THE TURBIDIMETER

(See Test Method C 115)²

6. Difficulties

6.1 Some operators have found it difficult to oscillate the tank the full 180° about a horizontal axis through the center of the tank. Complete rotation is necessary for proper agitation. A design of a rotatable holder is available on request from the Cement and Concrete Reference Laboratory at the National Institute of Standards and Technology, Gaithersburg, MD 20899.

6.2 A previously opened standard fineness sample will become unsuitable for calibration purposes. Whenever an unexpected difficulty is encountered, a new sample should be opened.

6.2.1 *Opening a Standard Fineness Sample*—Care should be taken when opening a new sample. The most recent fineness standard, beginning with SRM 114P, is packaged in plastic bags, which greatly simplifies opening the sample. Previous fineness standards were packaged in glass vials. When opening a sample packaged in a glass vial, contamination with glass chips shall be avoided. One satisfactory method of opening a vial consists of inverting the vial and pressing the sides in the empty section against a thin curved high-resistance wire mounted across the terminals of a small transformer.

6.3 The derivation of the Wagner equation is based on a light intensity (I_o) of 100 microamperes (μ A). For some unexplained reasons, the light intensities arrived at by calibrating with NIST SRM 114 have shown a great increase with some I_o 's being 150 or more. In addition to the calibration being time consuming and laborious, test results are subject to increasingly larger errors as the I_o deviates further from 100 μ A.

7. Care of Apparatus

7.1 Great care must be taken to keep the various parts of the apparatus clean, and to maintain good electrical contacts. This last precaution especially relates to the electric bulb, rheostat, and the wire leads to the shelf. It is particularly important to keep the water cell clean. At times the cell glasses will become dirty. A wavy film of growth will sometimes develop in the cell, causing decided fluctuations in the intensity of the transmitted light when the water is disturbed by the movement of the shelf. Keep the cell completely filled with distilled water. A drop or two of potassium dichromate solution added to the water in the cell will serve to inhibit possible algae growth but will not be enough to discolor the water.

7.2 The tank support must be so adjusted that, when using the specified volume of suspension the top of the suspension will be at the level of the zero mark on the graduated scale of

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the turbidimeter case, and the tank will be in a perpendicular position. When a new tank is put into use, check for proper elevation and adjust the tank if necessary.

7.3 D'Arsonval microammeters are very sensitive instruments, and are necessarily made up of very delicate parts. Observe the following care and handling instructions: (1) Use with care since pivots, jewels, and springs are subject to damage due to rough handling. (2) Do not place microammeters where they will be exposed to vibration or shock. One severe shock, such as hammering on the table on which the microammeter is sitting, can cause permanent damage. (3) Eliminate static charges and magnetic fields for the instrument and surrounding area. A static charge may be eliminated by breathing on the affected part. Do not place instrument on a table top of magnetic material or near a strong external magnetic field. This may be done inadvertently by placing the instrument on a metal table top or on the metal stand of a magnifier. (4) When taking a reading, very lightly tap the case to eliminate slight friction in the movement system. Hard tapping may damage pivots and jewels. (5) Store instruments in cases free from dust, acid fumes, excessive heat, strong magnetic fields, moisture and vibration. (6) Terminals of a microammeter should be shorted and, if available, movement locking devices should be used while the instrument is in storage or transit. (7) Always handle microammeters carefully and occasionally check the accuracy of indication.

7.3.1 A digital microammeter is a suitable replacement for the D'Arsonval type microammeter. Although this device has a higher internal resistance, the photocell response is linear in the normal operating range encountered in the turbidimeter.

7.4 Examine the timing buret frequently to guard against the presence of particles of lint, glass, and the like in the capillary. A filter made of 45- μ m (No. 325) wire cloth is recommended for the top of the buret, and a cover placed over the top of the buret when it is not in use. The dimensional requirements for the buret are shown in Test Method C 115, for Fineness of Portland Cement by the Turbidimeter.²

7.4.1 If a marked buret is not available an unmarked one may be used by placing a tape lengthwise along the barrel of the buret where the markings usually appear. Drainage times may be calculated if the viscosity of the kerosine is known, or the drainage times may be measured with a stop watch by timing the flow from a buret known to be correct. Lines are then marked on the tape corresponding to the measured drainage times.

7.5 Examine the stirring brush for dimensions and condition. Although there is no specified distance between the brush and the inside of the test tube, the distance is critical for stirring action. Restandardization is necessary when a new brush is installed or a different test tube is used for agitating the sample. The contour of the bottom of the stirring brush should match the contour of the test tube. Many stirring brushes when received do not meet this requirement and it may be necessary to trim the bristles in order to achieve this condition.

7.6 Many of the difficulties experienced with the turbidimeter stem from electrical troubles. (1) Corrosion can occur in electrical contacts and wires of rheostats. Some turbidimeters ground the light to the movable shelf through the reflector

housing and the wire from the power source is terminated with a spade connector fastened to the bottom of the movable shelf. This can be a hidden source of corrosion and looseness. (2) Connecting wires develop hidden breaks under constant flexing from raising and lowering the shelf. (3) Looseness in connecting wires, the light bulb and its socket, and the reflector in its holder can cause flickering or a reduction in light intensity. (4) Storage batteries should be kept well charged. (5) A light bulb, after prolonged usage, can become darkened and emit light of a different color or intensity which changes the instrument characteristics. (6) Regulated power supplies, which some laboratories use to replace storage batteries, are subject to malfunctioning and failures.

7.7 Do not use kerosene more than once. There are some brands of specially processed kerosines on the market. These are usually water white and have an odor differing markedly from the characteristic odor of customary market grade kerosine. **Caution**—Caution should be used in substituting specialties for the market grade of kerosine since differences in viscosity and density will influence results at other than the calibration value. If difficulty is encountered in calibration, a new supply of kerosine has been known to solve the problem.

7.8 It should be remembered that the basic premise of the Wagner turbidimeter is that the standard light intensity is adjusted to a value of 100 μ A. Current standardizing procedures "force" the I_o regardless of condition of the photocell or light bulb or a suspending liquid of incorrect density or viscosity. By setting the light intensity near 50 μ A with the filter out of position and then measuring the resulting intensity through the filter, the ratio can be used to calculate the theoretical I_o through the filter. If the theoretical I_o differs too greatly from the I_o determined in the standardization procedure, there is the possibility that considerable errors could creep into the calculation of fineness for high-fineness materials. The digital microammeter with its range of 199.1 μ A allows the theoretical I_o to be measured directly without using the above ratio method.

7.9 Linearity of response of the photocell is also basic to proper operation. As photocells age their response to higher intensity of light may decrease. When a new cell is installed, set the intensity of light with the filter removed at a value near 50. Also measure the intensity with the filter in place and record both values. After a cell is in use, to see if the response has changed, set the intensity through the filter at the observed value. After the filter is removed, a substantial decrease in value from the original reading indicates that the cell should be replaced. This drop in response, although not influencing results at or near the calibration value, will show a trend toward calculated high values for high fineness products.

7.10 The parabolic reflector should remain clean, bright, and highly polished. Cleaning may be accomplished by using lens tissue or a very soft, lint-free cloth. Avoid touching the reflector surfaces with the fingers. The lens over the reflector assembly should be clean and free from fingerprints. A lamp, after being placed in the socket, should be wiped clean.

7.11 When the lamp filament is at the focus of the mirror, the rays of light will be approximately parallel and the response of the photocell should be greatest. Turning the cell in the hood

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could vary the response. Rotate to obtain maximum response. Rotating the bulb will also aid in “peaking-in” before locking the bulb in position. When this has been accomplished, a piece of paper placed in front of the hood of the photocell will show an image of the slot in the shield. The edges of the spot will be reasonably straight and the image will have a reasonably uniform distribution of light.

7.12 The turbidimeter should not be located near a source of variable heat such as a radiator, hot plate, or autoclave. Good laboratory planning should include the turbidimeter location in the constant temperature room.

7.13 No light-colored wiring or shiny pieces of metal should be used in the interior of the turbidimeter cabinet. Black gaskets should be used to seal the ends of the water cell.

7.14 Both linseed oil and oleic acid change with age or by exposure to heat and light through oxidation. If difficulties arise in calibration, a fresh supply of dispersing agent should be obtained. Dropping bottles commonly used to dispense the agent should be kept free from the thickened jelly-like oxidation products. Many laboratories use a small 1 or 2-oz dropping bottle and keep a primary supply in a container which is not opened frequently.

7.15 Scratches on the face of the tank may affect transmission of light. The tank should be kept clean. An oily film accumulates on the interior tank faces. Removal is facilitated with a lint-free wiper, or a synthetic rubber “squeegee.” One product used successfully is a piece of lintless foam on a stick.⁵

FINENESS BY THE 45- μ m (No. 325) SIEVE

(See Test Method C 430)²

8. Spray Nozzle

8.1 Care of the spray nozzle is important. In areas where considerable lime is present in the water, the holes of the spray nozzle have a tendency to become plugged with lime which will alter the flow rate. When cleaning a nozzle, wires should not be used due to the possibility of enlarging the holes. It was recommended that dilute (10 %) hydrochloric or acetic acid should be used to avoid chemical attack on the brass from which the nozzles are usually constructed.

8.2 A spray nozzle is deemed acceptable if the flow rate is between 1500 and 3000 g/min at 10 psi (69 kPa). No single hole in the perforated plate should be large enough to accept a No. 74 drill (0.0225 in. or 0.57 mm). **Caution**—Caution should be used when checking hole diameters with a twist drill because all holes in the plate, except the center one, are drilled on an angle of either 5 or 10°, and if the drill is not inserted at the proper angle there is a possibility of enlarging the hole with the drill which is made from a much harder material.

8.3 *Care of Apparatus*—The cleaning procedure given in C 430 must be followed. Cleaning or rinsing with dilute hydrochloric or acetic acid solutions is to be avoided. Cleaning solutions are restricted to soap or detergent type solutions. Some laboratories have large ultrasonic cleaners for cleaning cube molds, etc. To use this apparatus it is suggested that the

sieve be immersed in the cleaning solution in a large beaker which in turn is placed in the large ultrasonic tank filled with the usual cleaner.

8.4 45- μ m (No. 325) *Sieve Cloth*—After considerable research into the reasons for high correction factors for the 45- μ m sieves available, it was determined by Subcommittee C01.25 on Fineness that the limits in Specification E 11, for Wire-Cloth Sieves for Testing Purposes,² are not tight enough to ensure a low correction even if Specification E 11 is met. Accordingly, if an operator has trouble in obtaining a reasonable sieve correction of 10 % or less, the reliability of the sieve should be checked using the “picket fence” approach discussed in Appendix X1.4 of Specification E 11. In order to check a sieve, a 350 line to the inch interference grating such as furnished by W. S. Tyler can be used. A piece of cloth with 45- μ m openings and average wire diameter will have 339 openings. If the number of openings is within ± 10 of the nominal 339, a sieve correction of 10 % or less should be obtained.⁶

FINENESS BY THE AIR PERMEABILITY TEST

(See Test Method C 204)²

9. Air Permeability Fineness Test

9.1 Details of this procedure are well covered in ASTM Test Method C 204, for Fineness of Portland Cement by Air Permeability Apparatus.² The following remarks supplement those instructions:

9.1.1 The U-tube manometer should be filled to the lowest mark with the manometer fluid. The liquid should be at this midpoint after any liquid on the side of the manometer has had sufficient time to drain. Immediately after a test, the liquid will be slightly below the mark, due to the fact that a small portion remains on the glass. This will not affect the results of a subsequent test. Slight variations in the height of this liquid may be noted at different temperatures. Do not attempt to adjust the quantity of liquid for different temperatures. Silicone stopcock lubricants should not be used with this apparatus. Each new lot of filter paper disks should be carefully checked for conformance with specification requirements.

9.1.2 When preparing the bed of cement in the cell the side of the cell should be tapped sufficiently to level the bed. This leveling should be confirmed by visual inspection, prior to placing a filter paper disc on top of the cement.

9.2 The latest revision of Test Method C 204 substitutes the quantity $(b - \epsilon)$ for the quantity $(1 - \epsilon)$ in the denominator of equations 5, 6, 7 and 8 of Section 6 and the value of $(b - \epsilon_s)$ or $(b_s - \epsilon_s)$ for $(1 - \epsilon_s)$ in the numerator of the same equations. The purpose of these changes is to eliminate inadequacies in the former porosity function. Data indicate that differences in the fineness test results with changes in porosity are considerable when using the former function. The new function nearly eliminates the differences caused by changes in porosity.

⁵ This item is marketed by Texwipe, 51 Prospect Place, Hillsdale, NJ 07642.

⁶ Electroformed sieve sheets usually have a sieve correction factor of less than 10 %. 45- μ m electroformed sieve sheets conforming to C 430 are available from: Buckbee-Mears Co. 278 E. 7th Street, St. Paul, MN 55101.

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9.2.1 The value of the constant, b , for a material other than portland cement can be determined from air permeability tests at different porosities (4 or 5 should be ample) on that material. Plot the value of $\sqrt{\varepsilon^3 T}$ on the horizontal axis against ε on the vertical axis. The porosity value, b , is the value of ε where a straight line drawn through the points intersects the zero $\sqrt{\varepsilon^3 T}$ value.

9.2.2 Although the 0.9 value has been determined for portland cement and new values determined by experiment for other materials, work in ASTM Subcommittee C01.25 on Fineness demonstrated that for blended cements, granulated blast-furnace slags, silica flour, and fly ashes, that the constant of 0.9 in the porosity function in all cases gave better correlation than the use of the unity constant.

TEMPERATURE AND RELATIVE HUMIDITY, STORAGE EQUIPMENT

10. Laboratory

10.1 ASTM standard test methods require that the temperature and humidity of the cement testing laboratory be maintained within controlled limits. This includes temperature limits for dry material, equipment, mixing table or slab, and mixing water. In general, increase in temperature is accompanied by decrease in setting time and usually by accelerated increase in strength. Drafts of air and low relative humidity will cause rapid evaporation of water from the mixes. Numerous laboratories have installed automatically controlled equipment for close control of room conditions. Since the temperature and relative humidity of the laboratory have a great effect upon the behavior of mixes, the temperature and humidity tolerances stipulated in standard test methods have been chosen to minimize that effect.

10.2 The laboratory temperature and humidity requirements have been developed for technical reasons and are an integral part of the testing system, and are not for the comfort of the cement tester.

11. Moist Cabinet and Moist Room

11.1 Specification C 511 requires that the temperature and humidity of the moist cabinet or room be closely controlled. Recirculating water sprayed through ducts, or water curtains flowing down inner walls, as well as fog sprays, have been used for maintaining the required humidity in cabinets and moist rooms. When fog sprays are used, precautions should be taken to prevent water falling upon the surfaces of the freshly molded specimens. Figure 1 in Specification C 511 is an example of a fog spray that combines water with air pressure to output a fine mist in a surprisingly large space. This design is excellent for a moist room and has been shown to not require as much cleaning as water sprays. Pans of water in the top of the cabinet, and pieces of wet wicking on inner surfaces of walls, may prove helpful in maintaining the necessary humidity by bringing large areas of wet surfaces into contact with the air. Numerous cabinets are available completely equipped with automatically controlled conditioning units. A temperature recorder (see Section 13) is required as a means of indicating the performance of the cabinet or moist room.

11.2 Moist cabinets and moist rooms that are exposed to surrounding air of substantially different temperature, require good insulation of the walls to maintain specified conditions. Plenty of water does not necessarily mean high relative humidity in a cabinet. If the necessary cooling of a poorly insulated cabinet is done by cold water, it may mean that the differential between the air and water temperatures will be such that the relative humidity will be less than the minimum relative humidity allowed. If refrigerator cooling coils are used, it is best to immerse them in the conditioning water system, because exposed coils tend to remove moisture from the air. Recirculation of water, when employed, must be sufficiently rapid to ensure that there is no great temperature differential between the water and the cabinet air.

11.3 The doors of the moist cabinet should be well-fitting and should be kept closed except during the actual operation of introducing or withdrawing the test specimens. It is desirable to have separate compartments for the time-of-set test specimens and the specimens for other tests, because of frequent opening and closing of the doors to make observations on the time-of-set test specimens. The shelves on which freshly molded specimens are placed should be level.

11.4 Specimens in storage should look and feel moist. When an area in a cabinet or room has specimens that look or feel dry, determine if:

11.4.1 The door has been left open,

11.4.2 The humidification has been shut off for a period of time,

11.4.3 The water spray(s) located in that area are functioning properly,

11.4.4 The temperature control is malfunctioning,

11.4.5 Cooling or heating for the area delivered as forced air is dry, or

11.4.6 Too many specimens are located in the area.

12. Storage Tanks

12.1 Specification C 511 requires storage tanks to be of non-corroding material. Test specimens should be completely immersed during the water storage period. Thermostatic control of the tanks or the room surrounding the tanks is required. This means that automatic control of both heating and cooling must be provided to maintain the temperature within the required limits. If the thermostatic control is in the tanks, then the probe for the controls must be located in the tank water. Placing the storage tanks in temperature-controlled moist cabinets, moist rooms, or other conditioned room without additional controls in the tank is acceptable.

12.2 The water in the storage tank(s) is required to maintain high-calcium hydrated lime saturation. A small cloth bag or sock of high-calcium hydrated lime placed in each tank has been shown to maintain this saturation. Periodically check the bag to ensure that the lime has not completely dissolved. This water should not be used as mix water in any test procedure. Storage water and tanks should be clean and free of any substance that might possibly affect the test specimens. The water should be changed as required. Continuous fresh running water, or demineralized water, has been known to lower strength results due to excessive leaching and should not be used in storage tanks. When the storage tanks are independent

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of moist cabinets or moist rooms, it is recommended that the tanks be equipped with a recording thermometer with its bulb in the storage water.

13. Temperature Recorders

13.1 Temperature recorders are required for moist cabinet and moist room, and also for the storage tanks if the latter are separate from the moist storage equipment. The specification requires that the temperature recorder record the temperature a minimum of every fifteen minutes and be accurate and readable to 1 °C. Check your temperature recorder to see if it is able to be used in moist air. If not, a remote probe type should be used.

13.2 A temperature recorder is required for each tank if it is not located within the moist cabinet or room. For the purpose of temperature recording, several tanks can be considered as one tank if it meets the following requirements:

13.2.1 All of the tanks are interconnected with tubing that allows the water to flow between the tanks,

13.2.2 Some means of circulation is provided between tanks, and

13.2.3 Temperature variation between the tanks must not exceed 1.0 °C when checked and recorded weekly.

MIXING CEMENT PASTES AND MORTARS

(See Test Method C 1005² and Specification E 617²)

14. Weights and Weighing Devices

14.1 The balance and the weights used in weighing the materials for the mixes should conform to the requirements of the respective standards, and should be kept clean and in good order at all times. Occasional check tests should be made, including the checking of any graduated beam on the scale. Appropriate accuracy requirements for graduated beams or dials on scales can be found in Test Method C 1005. Special single weights of sizes suitable to certain tests, such as 250, 300, 500, 750, and 900 g, may be used, both for convenience and to minimize the probability of errors in weighing the batches. Appropriate tolerances for weights between 1 and 1000 g, but not listed in the test methods, may be interpolated from the tables given in the methods. Weights should be checked occasionally and should be kept in blocks or other suitable containers to minimize wear. Pockets of weight containers should be kept free of sand and dirt. Balances should not be loaded beyond their capacity, which should be marked on the balance. Tops of balance tables should be plane to guard against balances easily sliding out of position.

15. Glass Graduates

15.1 The mixing water should be measured in a glass graduate of the capacity and type specified in the applicable test method. If not definitely specified, the capacity should not be more than twice the volume of water required in the mix. A 250-mL cylindrical graduate, reading in one direction only, is particularly recommended as suitable for use in the tests for normal consistency, tensile strength, time of setting, and autoclave expansion. Graduates should be checked to see that they conform to the requirements for accuracy of delivery. In some cases a very large error has been found—as much as

several millilitres. Graduates that have been tested and found satisfactory should be marked appropriately. Rubber guards around the tops of the graduates will greatly aid in reducing breakage.

16. Glass Burets

16.1 Some laboratories have found burets of the dispensing type, which discharge rapidly, very satisfactory for measuring the mixing water. However, such burets should be so mounted as to discharge directly into the mix, and not into an intermediate container which involves possible loss of water from the mix. The use of rubber delivery tubes with pinchcocks, which can readily shift from one position to another, may introduce appreciable error.

17. Testing Graduates and Burets

17.1 The National Institute of Standards and Technology *Circular C434*, “Testing of Glass Volumetric Apparatus,” gives information on specifications, special requirements, tolerances, and other matters relating to the testing of glass graduates and burets. The following information will be helpful to operators in calibrating water-measuring apparatus:

17.2 A temperature of 20°C (68°F) is regarded as standard for testing glass volumetric apparatus. The relation of height to diameter should be such that the graduation marks are not less than 1 mm apart and also that the graduated height is at least five times the inside diameter. Graduations should read in one direction only and should be of uniform width, continuous, finely but distinctly etched or engraved, and perpendicular to the axis of the cylinder. The main graduation lines of cylinders should extend entirely around the circumference and should be numbered. The least graduations should extend at least one seventh of the way around, and the intermediate graduations should extend at least one fifth of the way around. The numbers indicating the capacity of the graduate should be placed immediately above the marks to which they refer.

17.3 Graduates should be calibrated with water to deliver the indicated volume at 20°C (68°F). In all apparatus where the volume is limited by a meniscus, the reading is made on the lowest point of the meniscus. Apparatus must be sufficiently clean to permit uniform wetting of the surface, and the entire interior of the vessel should be wetted in filling. Apparatus must be allowed to drain a sufficient time before readings are taken.

17.4 In testing graduates and burets, the capacity of any interval may be determined by weighing the water delivered by the interval in question. The water is delivered into a weighing flask of convenient size provided with a stopper. The temperature of the water is observed either immediately before or immediately after delivery from the apparatus.

17.5 In making the weighings, it is both convenient and accurate to use the method of substitution. By this method a constant tare is kept on one pan of the balance, while on the other pan is placed the object to be weighed and with it sufficient weights to secure equilibrium.

17.6 Methods based on the transfer of water from a standard container, such as a buret, into the instrument to be tested can be utilized for items such as cylinders.

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18. Gloves

18.1 Rubber gloves should always be used when handling cement pastes and mortars. Gloves should be well-fitting and comfortable, and flexible enough to permit easy movement of the fingers.

19. Mixing Water

19.1 In general, water fit for drinking is satisfactory for cement testing. In case of doubt, distilled or deionized water may be used for comparison or reference. The amount of water present in a mix is of great importance, particularly with regard to the setting time and strength. Water much in excess of the required quantity will prolong the setting time and lower the strength. Less than the proper amount of water may shorten the setting time and result in strength variations from the normal, the latter sometimes being due to the difference in molding properties of the mortar. The amount of water used is expressed in millilitres, is usually measured by volume, and must be carefully measured.

20. Time of Mixing

20.1 A timer, preferably one with a sweep-second hand, should be so located that the operator can conveniently observe the time.

21. Size of Batch

21.1 The size of the batch is closely related to the possible thoroughness and uniformity of mixing, other conditions being the same, and is therefore not without its effects on the results of the tests. Small batches may lose relatively large amounts of water by evaporation. On the other hand, with the same amount of time and mixing, much larger batches may not be so thoroughly or so uniformly mixed. Moreover, the longer time required to mold the larger batches into specimens may result in considerable evaporation of water, stiffening of the cement through setting action, and an accompanying decrease in the ease of molding and in the uniformity of the results.

22. Mechanical Mixing (See Practice C 305)²

22.1 *Mixer*—The mechanical mixer should be inspected frequently, and kept clean and oiled. Care should be taken to prevent leakage of oil from the gear housing falling into the bowl during mixing operations. This will change the consistency and greatly affect the tests. Before the bowl is raised to the mixing position and the mixing operation started, a check should be made to ensure that both the paddle and bowl are firmly seated. Care should be taken in removing the paddle from the mixer, as the bowl can be easily dented if the paddle is dropped. If dents appear in the bottom of the bowl, they should be removed. Filling the underside of the raised portion of the bowl bottom with solder reduces denting, thus increasing the useful life of the bowl.

22.2 *Bowl Clearances*—Proper paddle to bowl clearances are necessary to make the tests accurate and reproducible. When the clearance is too great, nonuniform mixing results. When the clearance is too tight, grinding action reduces the size of the standard sand, which changes consistency and results. The tight clearance also causes excessive wear of the

paddle and the bowl, necessitating frequent replacement. Small adjustments can be made by use of the adjustment bracket specified in Practice C 305. Major adjustments can be made by raising or lowering the motor housing.

22.2.1 There are several important factors in monitoring the specified clearances between the paddle and the mixing bowl. The assembly, shaft and paddle should be visually inspected for wear each time clearances are checked or adjusted.

22.2.1.1 The bushing, which holds the paddle shaft in place in the paddle gear assembly, must be properly snug during operation. By moving the paddle-shaft assembly by hand, the condition of the bushing can be determined. This hand movement of the paddle-shaft assembly should only be checked with the power cord to the mixer disconnected. Remove the bowl from the mixer and place the paddle on the shaft. Try to move the paddle by hand with a push-pull motion toward the center of the assembly. Movement or eccentric “play” in the assembly above the shaft indicates a worn bushing.

22.2.1.2 If the shaft remains still, while the paddle visibly moves during the hand movement, then three possibilities should be considered: the shaft has become worn and unable to properly support the paddle during mixing; the pin holding the paddle in position on the shaft has become worn and is no longer able to retain the paddle in the correct position to achieve the specified clearance; or the bore hole in the paddle has become enlarged and the paddle will no longer fit snugly on the shaft.

22.2.1.3 Another potential problem is the spring on the paddle shaft which holds the paddle in place during mixing. The spring may be damaged over a period of normal use. This seldom occurs but should not be overlooked. A damaged spring will be especially noticeable during the mixing procedure.

22.2.2 It is recommended that a set of tolerance gages be available for checking the critical bowl to paddle clearance. These gages can be made from the appropriate size drill rods. Long-shanked Allen wrenches of the proper size can also be used. Three sizes are required as described in the Apparatus section of Practice C 305.

22.3 *Markings of Bowls and Paddles*—Since most bowls and paddles are not quite identical, once a set has been adjusted for clearance, it should be marked for identification.

22.4 *Procedure*—The following are some of the conditions that can cause error:

22.4.1 Incomplete drying of the bowl and paddle between tests,

22.4.2 Excessive heating or cooling of the bowl and paddle during washing, which will affect consistency,

22.4.3 Failure to follow correct sequence of mixing operation, and

22.4.4 Loss of material during addition. (The introduction of sand into the mixer bowl can be facilitated by the use of a funnel.)

22.5 In any case requiring a remixing interval, any mortar adhering to the side of the bowl shall be quickly scraped down into the batch with the scraper prior to remixing.

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23. Hand Mixing

23.1 The requirements for hand mixing have been deleted from the ASTM specifications for portland cement. The procedures may be useful for research and special purposes.

23.2 *Mixing Slab*—The height of the mixing table slab above the floor may have a great influence on the efficiency of hand-mixing and molding operations. A height of about 36 in. is correct for the average operator. For unusually tall or unusually short operators, the height of the table top should be adjusted accordingly. The slab should be plane and made of nonabsorbent material. Steel plate at least $\frac{1}{4}$ in. in thickness, or plate glass about $\frac{3}{4}$ to 1 in. in thickness, is recommended for the tops of mixing tables. The mixing table should not be subjected to undue exposure to sun and drafts, or be placed too near radiators, hot plates, steam apparatus, etc. The slab must be kept clean and free of cement and oil. Glass plates are frequently chipped and steel plates nicked by the practice of tapping the trowel corners edgewise and sharply downward on the slab to remove mortar from the trowel. Placing unusually hot or cold objects on glass mixing slabs may crack the glass.

24. Mixing of Cement, Sand, and Water

24.1 In some cases the specifications require that the batch must be thoroughly mixed dry, on the slab, prior to the introduction of the water. The materials should not be scattered widely, but should be neatly and thoroughly mixed by turning with the trowel and by rubbing them lightly on the mixing table with the hand until the mass shows by its uniform appearance that the ingredients are well mixed. This method will cause less segregation than rolling the dry materials on a paper. A crater which will hold the entire desired quantity of mixing water without overflow or loss should be then formed in the dry materials.

24.2 The mixing water must be added exactly as required by the test method. When mixed by hand on the slab, the whole of the mixing water must be poured at one time into the crater in the dry materials. The dry materials should then be turned, as quickly as possible, from the outside into the crater by means of a trowel, covering all the water in not more than 30 s. It should be noted that this 30-s interval for covering is the maximum time allowed for this operation. A skilled operator will usually finish covering in 15 or 20 s, and then the timing of the 30 s for absorption should be started immediately. The absorption time is always 30 s, regardless of the time required to complete the covering operation. For example, if the covering is completed in 15 s, the total elapsed time for the two operations, covering and absorption, should be 15 + 30, or 45 s.

24.3 The amount of water in a mix may be unintentionally increased by using wet gloves, or by pouring the dry materials onto a wet slab. Loss of water may be somewhat avoided by mixing the materials and the water so as to spread as little as possible of the mix over the surfaces of the gloves or slab.

NORMAL CONSISTENCY

(See Test Method C 187)²

25. Vicat Apparatus

25.1 The Vicat apparatus should be inspected frequently and kept clean, free of rust, and oiled. The plunger and needle ends should be free of oil or grease during a test. The weight and diameter of the plunger assembly should be checked to determine whether they conform to the requirements of ASTM Test Method C 187, for Normal Consistency of Hydraulic Cement.² The plunger should be removed from the frame and weighed with the proper assembly of parts. Some Vicat apparatus are equipped with separate weights for use with needle or plunger. The plunger and needle diameters should be measured with a micrometer at the end and at several points over their penetration portions. The plunger and needle should be longer than the height of the mold, and must be straight with end surfaces flat and perpendicular to the axis. The edges should be true and sharp and the surfaces free of rust. The plunger should be clean, lightly oiled at contact with the frame, and should slide freely through the guides. If plunger and needle assembly require considerable adjustment, weights should be rechecked after such adjustments have been made.

25.2 The top and bottom inside diameters of molds, as well as height of mold, should be measured at several points.

26. Test Method

26.1 For details of mixing in the determination of normal consistency, see Test Method C 187. If a trial paste is too dry, it should not be remixed with more water but should be discarded, and a new sample should be taken in order to avoid error resulting from evaporation and other causes. The tossing referred to in Section 6.2 of Test Method C 187 is comparable to tossing a ball from one hand to another with the free travel of the ball being approximately 6 in. (150 mm). The tossing of the paste from hand to hand is considered to be one of the most effective methods to form the paste into a spherical shape, enabling easy insertion into the Vicat ring, with a minimum amount of additional sample manipulation.

26.2 After the correct Normal Consistency has been determined, the test specimen may be used for the Vicat Time of Set test, the remaining paste may be used for the Autoclave test and the Gillmore Time of Set test.

27. Care in Determination

27.1 The accuracy of the normal consistency determination depends upon the proper mixing and placing of the cement paste in the mold, removal of the surplus with as little disturbance of the mass as possible, freedom of the apparatus from vibration, and careful manipulation throughout the test. Care should be taken to center the test specimen under the plunger. Take initial reading and release plunger immediately. The time for operations performed between the end of mixing

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and release of the plunger shall not exceed 30 s. Care must also be taken to avoid shaking the Vicat apparatus when releasing the plunger and to avoid leaning upon or shaking the table during the test. The operator must possess considerable skill and dexterity in order to make this test satisfactorily. It is a very important determination, however, because upon it depends the percentage of water that is to be used for certain other determinations.

AUTOCLAVE EXPANSION

(See Test Method C 151 and Specification C 490)²

28. Autoclave

28.1 If the autoclave does not meet the specified heating rate, one of the following may be the cause:

28.1.1 Low input voltage, or

28.1.2 Need of a new heating element.

28.2 If the autoclave does not meet the specified cooling rate, the cooling can be increased by providing more ventilation for the autoclave and by allowing a fan to blow on the autoclave after the heat is shut off. An accumulation of dirt in the space between the pressure chamber and the outer shell may retard cooling.

29. Use of Thermometers

29.1 The section on Safety Precautions of Test Method C 151 for Autoclave Expansion of Portland Cement, requires that a thermometer always be used, together with the pressure gage, so as to provide a means of detecting any failure of the pressure gage to act properly and to indicate any unusual conditions. The thermometer is to be placed in the thermometer well which is specified in Test Method C 151. Neither mercury nor asbestos shall be used in the well due to health hazards. Oil is not recommended for use in the well. If a laboratory considers that conditions require the use of some liquid in the well, use might be made of one of the liquid organic phosphates of the type used as plasticizers in the plastic industry. Dow Plasticizer No. 5 has been found satisfactory for temperatures above that prescribed for the autoclave test in Test Method C 151.

30. Reference Bar

30.1 The steel reference bar mentioned in Specification C 490 should be used at least at the beginning and end of the readings made within a half day. Such use of the reference bar may seem like an unnecessary refinement for routine tests, particularly in those cases where test specimens are measured in temperature-controlled rooms. Nevertheless the use of the reference bar will guard against errors caused by unsuspected changes in position of the dial, and will permit measurements to be made even when the comparator may have undergone adjustment or replacement during the autoclave test. After the bar is put into use, the ends should not be rubbed with abrasives, but should merely be wiped with a cloth. The ends should be protected against corrosion or injury, and the length of the bar should be carefully determined occasionally to detect any appreciable change. The bar should be so marked that the

same end can always be kept uppermost when the bar is used. The central 4 in. (100 mm) should be covered with a rubber insulating grip.

31. Rupture Disk and Vent Valve

31.1 In Test Method C 151 a rupture disk is the pressure relief safety device specified for use with the autoclave used in this method. State and local regulations should be checked regarding the use of rupture disks for this purpose.

31.1.1 Care should be taken in selecting the disk material. Of the materials available for making disks, Inconel has the flattest temperature/pressure curve retaining approximately 93 % at 420°F (216°C) of the rated burst pressure at 68°F (20°C). The holder material for the disk may be either carbon steel or stainless steel. Disks may have a relief area of 0.5 to 1 in. A 1-in. relief is considered more than adequate. The manufacturer should be consulted about the particular application.

31.1.2 Rupture disks cannot be tested without destructive bursting. A periodic inspection/replacement is recommended at least yearly as a method of preventive maintenance. If there is any reason to suspect corrosion or the autoclave is used heavily, a more cautious approach should be taken by replacing the rupture disk every six months until a history is established. Then, if there are no problems, an annual replacement may be used.

31.1.3 There are applications in industry where a rupture disk is placed in line ahead of the relief valve as protection against corrosion. Although this arrangement does not appear in Test Method C 151, some manufacturers recommend the practice to ensure an operable valve at all times while reducing maintenance.

31.1.4 The American Institute of Chemical Engineers in their publication 54-B, "Selection of Rupture Discs for Emergency Relief of Chemical Process Equipment," describes the sizing, selection, and installation of rupture disks. This publication is recommended for those designing or operating a high-pressure steam vessel used in cement testing.

31.1.5 Rupture disks are actually differential pressure devices. If a disk is rated to burst at 100 psig, it will burst at 100 psig process pressure, only if the downstream pressure is 0 psig. If the disk is back loaded to 50 psig, it will not rupture until the process reaches 150 psig. It is important, therefore, to ensure that no pressure pockets can build in the piping between double-disk assemblies or disk-relief valve assemblies. This is generally accomplished by drilling the hold-down flange and installing an excess flow valve, pressure gage, or combination of the two. There is an ASME code requirement covering this matter.

31.2 During the early portion of the heating period, the vent valve is left open to permit air to escape from the chamber. A rubber tube, one end of which is attached to the vent and the other end of which is submerged in a beaker of water, will serve to indicate when the escape of the air is completed. A few drops of kerosine placed in the vent valve about once a week will aid in keeping the needle clean and in good working condition.

31.3 **Safety Precautions**—Experience has prompted Committee C01 to express the desire that publicity be given to the

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safety precautions that should be observed when operating cement testing autoclaves. Safety Precautions in Test Method C 151 will alert the operator to potential safety hazards.

32. Cooling of Test Specimens After Autoclaving

32.1 Test Method C 151 specifies that upon removal from the autoclave at the end of the test period, the test specimens should be placed in water the temperature of which exceeds 194°F (90°C), after which the water in the bath is to be cooled to the measurement temperature at a uniform rate by the addition of cold water. A cylindrical container approximately 8 in. (200 mm) in diameter and 16 in. (400 mm) in height, equipped with a bail, makes a very convenient chamber for the cooling operation. In use, the container is filled with water and placed on a hot plate at the end of the 3-h autoclaving period. Later, when the autoclave is opened, the rack and bars as a single unit may be lifted from the autoclave by means of a suitable metal hook and immersed in the near-boiling water in the container. The container may then be transferred to a sink, and cold water introduced through a rubber tube connected to a cold water spigot. The rate of cooling may be controlled by regulation of the flow of cold water through the tube.

32.2 It is to be noted that the temperature of heated tap water is seldom greater than 194°F (90°C).

TIME OF SETTING

(See Test Methods C 191 and C 266)²

33. Vicat and Gillmore Needles

33.1 The cement specifications permit the use of either the Vicat apparatus or the Gillmore needle, as specified. Weights of needle assemblies, and the straightness and diameters of needles should be checked frequently to determine whether they conform to the respective requirements of the applicable ASTM test methods; see also Section 25 of this manual. It is sometimes found that even new needles are not standard either as to weight or diameter.

34. Method of Making Gillmore Specimens

34.1 The method of making Gillmore test specimens is described in Test Method C 266.² The shape and size of the pats should be as specified in Test Method C 266, and the pats should be formed as shown in Fig. 1 of this manual. Clean, dry, unoled, plane glass plates should be used. There should be just enough troweling to give the proper shape and a smooth surface. The specified thickness should be observed closely. An overtroveled surface or a rough surface may materially affect the apparent time of set. The top of the pat should be brought to a smooth horizontal surface by a single stroke of the trowel. The flat surface of the pat should be parallel to the surface of the glass.

35. Storage of Specimens

35.1 Specimens should be stored in the moist closet or moist room during the determination of time of setting. It is usually necessary to take the specimens from the moist storage when a determination is being made. However, they should not be left

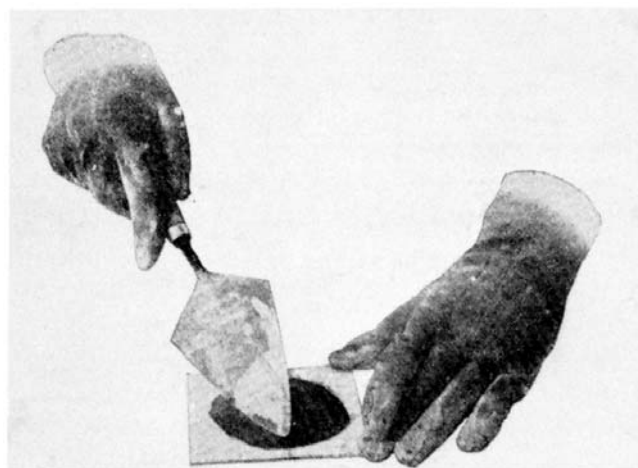


FIG. 1 Correct Method of Forming Cement Pat

in the air of the laboratory but returned to the moist closet as soon as possible after the trial test has been made.

36. Test Method

36.1 The surface of the test specimen must be well lighted throughout the test. The needle must be held vertical and applied perpendicular to the surface without shock. This is especially difficult in the case of the Gillmore needle, and great care is necessary. The use of a frame, as illustrated in Test Method C 266, will assist in this regard. In determining the end point, the needle, if not applied perpendicular to the surface, will give a crescent-shaped mark some time after the setting time should have been recorded.

37. Early Stiffening of Hydraulic Cement (Mortar Method) (Test Method C 359)²

37.1 Test Method C 359 is a mortar test for early stiffening, and there are no requirements for this test in standards such as Specification C 150, Specification C 595, or Performance Specification C 1157. It was originally developed for use in research on methods for determining the potential false set of hydraulic cements. While the paste method (Test Method C 451) gives an indication of the differences between false set and flash set, the mortar method gives more data for the user to make a decision when faced with early stiffening problems.

37.2 The mortar method has not found a lot of acceptance; since there is no specification requirement and it does not give adequate guidance as to evaluation of the results. Those who use this method in troubleshooting field problems involving early stiffening have reported better correlation than with the paste method. The evaluation (early stiffening rate and early stiffening recovery) shown in Test Method C 359 does not state what these values represent or how they relate to false set or flash set.

37.3 In earlier studies, Norris⁷ developed a method which calculates an early stiffening coefficient and a plastic recovery

⁷ Norris, David, Inter-laboratory Study to Establish Precision Statements for ASTM C359, Early Stiffening of Hydraulic Cement (Mortar Method). This paper has been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR: C01-1008.

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coefficient. The sum of these two coefficients is an index which can range from 0.00 to 2.00, with 0 indicating no false or flash set, 1.00 indicating pure false set, and 2.00 indicating pure flash set. The sums between 0 and 1.00 show varying degrees of false set, with 1.00 being the most severe. The sums between 1.00 and 2.00 show varying degrees of flash set, with 2.00 being the most severe. Examples of these and the methods of calculation are shown in the following table:

Data	NONE	FALSE	FLASH
Penetration—Initial	49	49	49
Penetration—5 min	49	0	0
Penetration—8 min	49	0	0
Penetration—11 min	49	0	0
Penetration—Remix	49	49	0
Max Sum = 3x initial = 147	147	147	147
Sum of 5, 8, and 11	147	0	0
Early Stiffening Coefficient (Max Sum-Sum 5, 8, 11)/ Max Sum	0.00	1.00	1.00
Plastic Recovery Coefficient (Initial-Remix)/Initial	0.00	0.00	1.00
Index = ESC + PRC	0.00	1.00	2.00

STRENGTH TESTS (GENERAL)

38. Storage of Specimens in Moist Closet or Moist Room

38.1 It is intended that the molds containing test specimens should be stored so that the upper surfaces of the specimens are freely and uniformly exposed to the atmosphere of the moist closet or room. Molds should not be piled one upon the other in such a way that the plates of the upper molds exclude air from the specimens beneath. The specimens should be kept in moist storage until they have attained the age of 24 h, when they are to be placed in the storage tanks. In no case should they be removed from the molds until they are at least 20-h old. If removed from the molds before they are 24-h old they should be replaced on the shelves of the closet until the expiration of the 24-h period. The removal from the molds should be done with care and with as little disturbance as possible, as specimens at the end of 24 h are not very hard and rough treatment may produce injuries which, although not apparent, will nevertheless affect the strengths.

38.2 When the freshly molded specimens are placed in the moist storage, each mold should be accompanied by an identification tag. On no account should identification depend on the position of a mold on the shelves or its proximity to some identified mold.

38.3 When the specimens are removed from the moist storage, it will be found very desirable to mark the reference numbers of the bottom faces of the specimens, after the plates have been removed but while the specimens are still in the molds. A graphite crayon, when used on these oily bottom surfaces which have been in contact with the mold plates, will form very distinct and durable markings. Colored crayons are not recommended because their marks may quickly fade under

the circumstances. The practice of using a sharp tool to scratch identification marks on the surfaces of mortar specimens is objectionable.

39. Precautions to be Observed in Testing Specimens

39.1 Specimens, except those to be tested at 24 h, must be tested as soon as removed from the storage water. Specimens to be tested at 24 h should be taken directly from moist storage and tested at once without previous immersion in water. When several 24-h specimens are to be tested at one time, it is recommended that they be removed from moist storage, placed in a pan, and kept covered with a moist cloth until ready for test. Indications of injuries, defects, or unusual appearances should be noted. When two or more specimens, other than 24-h specimens, are to be tested at one time, they should be removed from the tanks, placed immediately in a pan of water, and kept there until actually tested. As it is important to prevent changes in temperature, it is recommended that specimens be covered with water from the storage tank.

TENSILE STRENGTH

(See Test Method C 190)²

40. Briquet Molds

40.1 Briquet molds must be of the form specified in Figs. 1 and 3 of Test Method C 190, for Tensile Strength of Hydraulic Cement Mortars, and should meet the requirements for all dimensions given in Section 4.5 of Test Method C 190. Molds should be frequently checked to determine whether they conform to the tolerances specified. A three-gang mold is the most convenient to handle. Clamps should be of a type which will close the joints in the molds completely so as to ensure briquets of specified width (see Fig 1 of Test Method C 190). There should be enough metal in the sides of the molds to prevent distortion during use. The molds, well cleaned, should be examined to see whether the halves are properly mated and the clips are of such type and condition as will hold the halves tightly together. The widths at waist line should be measured, preferably and most readily, with “go and no-go” gages.

40.2 Compare the molds with the standard outline of briquet. Considerable difference in outline near ends may be encountered, which is important in case weights of briquets are being compared. The mold outlines should conform closely with the standard at the points of contact with the rollers of the testing machine grips. As these contact points will be about ½ in. (13 mm) above and below the center line of briquets, the briquet mold outline should compare closely with the standard in these regions.

40.3 The greatest thickness of each half of new molds should be measured at points between inner and outer edges at the waistline of the briquet, opposite each briquet space.

40.4 The molds should be cleaned soon after the hardened mortar test specimens have been removed; otherwise, the cleaning will be more difficult. Brushes having bristles made of brass, fiber, or other material softer than the molds are recommended for use in cleaning briquet molds.

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41. Testing Machines

41.1 The type of testing machine should be such as to conform strictly with the requirements of Test Method C 190 as regards rate and uniformity of application of the load and other details. The clips should be provided with rollers $\frac{1}{2}$ in. (13 mm) in diameter, with full bearing of 1 in. (25 mm) on the briquet, and mounted so as to permit easy turning during the test. (See Fig. 2 of Test Method C 190.) Many laboratories do not observe the latter detail sufficiently, with the result that eccentric strains may be produced in the briquet and erratic results may be secured. Moreover, the distance between the rollers in the clips should not vary appreciably, and the clips should be so designed as to keep the rollers in parallel positions during the test. The bearings of the rollers should be well lubricated. Petrolatum or cup grease will be found quite effective. Grit in the bearings or lack of lubrication often results in a jerky or nonuniform operation of a machine, and may produce undue stresses in a specimen. Long use will sometimes so roughen or deform the rollers that they must be replaced. Daily balancing of the beam is a good habit to acquire. The testing machine should be thoroughly cleaned before examination. Knife-edges and bearings should be inspected. Stirrups and knife-edge bearings should be free of nicks, dents, or worn spots. Knife-edges should be straight, sharp, and tightly held in beams. The various parts of the machine should be adjusted so that the beam balances and swings freely at zero load. The method of balancing the machine will vary according to the type and model. Individual parts of the machine should be calibrated as follows:

41.2 Check grips with respect to condition, lubrication, dimensions, and spacing of rollers. (See Fig. 2 of Test Method C 190 for details of important dimensions.) The rollers should be 0.50 ± 0.01 in. (12.7 ± 0.3 mm) in diameter and should be approximately $1\frac{1}{8}$ in. (28.6 mm) in length. The distance between the roller faces should be 1.25 ± 0.02 in. (31.8 ± 0.5 mm), and the rollers should be parallel within 0.02 in. and should be in the same horizontal plane. The bore of the rollers should be straight and the rollers should bear on the pins throughout their length.

41.3 Check pivots with respect to center of briquets when in grips, particularly with respect to the front and back of specimen.

41.4 Make at least three determinations of the rate of shot flow, using not less than 30-s intervals and timing by the second hand of a watch. The result should conform to the tolerances of the specifications. Inability to secure sufficient flow rate may be due to dirty or badly worn shot or to stoppage in outlets. A small quantity of flake graphite will assist in preserving the shot and facilitating flow.

41.5 Load tests may be made by means of a lever, or by dead weights placed upon a support suspended from the upper grip of the machine either by a saddle straddling the base of the machine or by a rod. In the latter method a metal briquet is placed in the upper grip and a rod, passing through the base of the machine, is screwed into this briquet. A support sufficiently large to hold test weights is attached to the lower end of the rods. It is necessary to remove the lower grip and "take-up" gearing to accommodate the rod.

41.6 A small, good-grade spirit level, firmly secured to the top of a test lever near the fulcrum before balancing the beam, may prove preferable to the separate reference pointer. The weight of the level must be included as part of the weight of the lever assembly.

41.7 Loads should be applied in increments of not more than 100 lbf (445 N) and the weight of shot required to counterbalance each load carefully determined. Make several determinations of the amount of shot required to balance the machine at each loading. After removing the loading lever or other device, ascertain what load indications are obtained with the amount of shot required for each of the loadings used. If the machine has both a spring scale and graduated beam device, record the readings by both devices. If the indicated loads are not within the tolerance, examine the various weights of the machine for discrepancies. If the error is constant for the various loadings, look for alterations in the machine, defective counterweights, and see whether proper balance was obtained at zero load. If the error varies, examine bearings, rider weights, and spring scale weighing device. The latter may be tested for irregularity in operation or graduation of dial by applying small increments of load and determining whether the indicated dial readings are proportional to the load applied. The weight of the test lever, suspension devices, etc., must be taken into account when calculating the test load. The dead weights should be accurately known and not assumed to be exactly as indicated.

41.8 If the testing machine cannot be corrected within the specified tolerances by minor adjustments, it is recommended that it be returned to the manufacturer for proper adjustments.

42. Molding of Briquets

42.1 The mold plates should be of sufficient thickness to prevent bending during molding and handling of the briquets.

42.2 It is recommended that the thumb pressures be applied in groups of two per briquet on each of six trips along the mold. Care should be taken that the thumb pressures are applied entirely to the surface of the mortar, and not to the surface of the mold. The specified thumb pressure of between 15 and 20 lbf (67 and 89 N) may be measured by molding the briquets on a platform scale or on other suitable apparatus that will indicate the pressure exerted. In the smoothing operation, the mortar should be finished flush with the top surface of the mold, using the flat bottom surface of the trowel to smooth the mortar, and exerting not more than 4 lbf (18 N) pressure. The trowel should be held so that the long axis of the blade is at a right angle to the length of the mold. Sand grains should not be allowed to remain on the face of the mold to which the plate is to be applied, as this may result in thick specimens and also may cause a loss of water between the mold and plate. Care should be taken to see that the briquets when furnished are exactly flush with the surface of the molds. The joints of the molds should be tightly closed in order to ensure specimens of correct width. Many operators find that a slightly curved trowel is of great assistance in making briquets because of the ease with which it can be used to clean off the mold faces and smooth the mortar within the molds. Only mineral oil should be used in oiling molds and plates.

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43. Precautions to be Observed in Testing Briquets

43.1 Briquets must be carefully centered in the clips; otherwise large variations in the stresses and consequently in the results are probable. Briquets should be inserted in clips so that the smooth surfaces that have been in contact with the mold plate are turned toward the back of the clips and placed in contact with the guide bar at the back. Bearing surfaces of the briquets and of the rollers should be clean, smooth, and free of sand or grit, and the rollers should be well lubricated.

43.2 After the briquet is properly centered in the clips, just enough load should be applied by hand, before starting the mechanically applied load, to take up the play of the machine and prevent jerky application of the load. Very weak briquets may not permit taking up the play of the machine, and must be handled carefully. Adjustments of the load control mechanism are often needed and at least daily trials should be made before tests are commenced, to determine whether the load is being applied at the specified rate of 600 ± 25 lbf (2670 ± 111 N) per min. A quantity of flake graphite added to the shot hopper will aid the smooth and uniform flow of the shot.

COMPRESSIVE STRENGTH

(See Test Method C 109)²

44. Cube Molds

44.1 Preparation and maintenance of cube molds can affect strength results. Variation in the strength test result can occur if the bearing surfaces of a test specimen are not plane. This condition can result in not loading the complete surface area of the cube with the end result being an abnormally low strength. Another variation can occur if water is lost from the specimen during curing. Both issues can often be traced directly to the preparation and maintenance of the molds used.

44.2 Molds should be cleaned promptly after cubes are removed. When cleaning cube molds, care should be taken to preserve the planeness of the interior faces. It is recommended that steel brushes not be used on brass metal molds. Brass molds should be cleaned with brushes of brass or fiber bristles. Before assembling the two halves of the mold, make sure that no sand or mortar particles exist on the contact surfaces, in the alignment mechanism, or locking mechanism. A build-up of debris in any of these areas can prevent the two halves from completely joining together. Significant build-up may result in problems in the interior of the mold, causing nonparallel opposite faces and excessive width in the molded specimens.

44.3 The molds and base plates should be coated with a thin coating of mold release agent. Molds should be wiped with a clean rag to remove any excess mold release agent. The standard requires that the contact surface between the two halves of the mold be sealed with light cup grease. The amount of grease should be sufficient to extrude slightly when the two halves are tightened together. This extrusion should be wiped clean from the interior surfaces of the mold. The standard permits the use of petroleum jelly to seal the mold to a clamped base plate. After clamping, there should be no grease or oil on the interior faces or in the bottoms of the molds. When sealing the molds to the base plates with wax, care should be taken to seal the contact surface between the mold and the plate from

outside the mold. Inspect the mold for any wax that may have inadvertently been placed inside the mold. This wax must be removed prior to molding the test specimens.

45. Compression Testing Machine

45.1 The machine should be periodically tested to determine whether the error in load indication, over the working range involved in testing the 2-in. (50-mm) cubes, is within the 1 % allowed by Test Method C 109. Compression testing machines should be frequently inspected, and the following are among the items that should be checked (not all of these items are applicable to all machines):

45.1.1 *All Machines*—smooth application of load, surface of lower platen protected by use of lower bearing block, power switch within easy reach of machine operator, availability of manufacturer's literature and drawings relative to the machine, information as to time and results of the last verification of the machine, and proper fuses on power line.

45.1.2 *Screw Loaded, Beam and Poise Machines*—weighing levers in proper positions and free-moving parts of machine, proper adjustment of counterweights on weighing beam, loading screws well lubricated and straight, ability to balance machine at zero load, and clutches and gear shifts in good working order.

45.1.3 *Hydraulic Machines*—freedom of movement of gage hands, proper “gag” in machine fitted with hydraulic capsule weighing system (gap should be measured at least once each month), ample oil in hydraulic loading system, hydraulic systems free of air, and proper operation of automatic cutoffs.

45.2 Compression machines should be regularly lubricated. Lack of proper lubrication of loading screws may result in “jumpy” application of load. A mixture of grease and flake graphite has been used successfully for this purpose. This mixture should be well brushed into the threads, the head of the machine being moved up and down during the lubricating process.

46. Bearing Blocks

46.1 Particular attention should be given to the planeness of faces of the bearing blocks, their diameter, and the proper centering and freedom of movement of the upper, spherically seated bearing block.

46.2 The test method requires that the bearing surfaces of the blocks be plane to 0.0005 in. [0.013 mm] when new and 0.001 in. [0.025 mm] when used. To determine the planes of the bearing surfaces of both blocks a machinist straightedge and 0.001 in. [0.025mm] feeler stock as shown in Fig. 2 is used. Planeness is critical since the entire surface of the cube should be in contact with the bearing surfaces of the blocks during testing. Less than full contact between the bearing blocks and contact surfaces of the cube may result in lower reported strengths.

46.3 Oversized spherically seated bearing blocks may also result in partial loading of the specimens. Spherical blocks are meant to lock in place after contact. When a block is significantly larger than the diagonal of the specimen, it is difficult to determine if full contact with the specimen is being made as the

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FIG. 2 Determining the Planeness of the Bearing Surface with Machinist Straightedge and 0.001 in. [0.025 mm] Feeler Stock

load is applied. For this reason, the test method limits the size of the upper spherically seated bearing block to a maximum of $3\frac{1}{8}$ in. [79.4 mm].

46.4 Proper centering of the cube is another possible source of variation in test results. Compression testing machines are designed to give the most accurate results when specimens are tested with the load applied through the center point of the machine. The test method requires that at least one of the diameters of the two bearing blocks used be close to the measured diagonal of the cube. This block is required to be used to center the cube in the machine. It further requires that the block being used in centering the specimen be fixed to the center axis of the testing machine.

46.5 The upper spherically seated bearing block should be checked for ease of movement prior to use. The spherical block is designed to account for variation in degrees of parallel between the two bearing blocks. A block with no lubrication or with an accumulation of dust, dirt or debris from test specimens, can lock in a non-parallel position or be difficult to tilt. Less than full contact with the specimen can affect test results. If the spherical portion of the block is difficult to move in its socket, it must be cleaned and lubricated before testing.

46.5.1 When cleaning and lubricating the spherical block, it will be necessary to separate the socket portion assembly from the spherical portion of the block. Wipe the surface of the sphere and the socket with a clean rag to remove any accumulation of dirt or debris. An example of a disassembled block is shown in Fig. 3. Apply a thin film of oil to the surfaces. If the block is removed from the machine, replace it in the center of the upper platen.

46.6 The test method requires that the bearing face of the upper spherically seated block lie in the center of the sphere. Conformance to this requirement is determined by measuring the overall height of the spherical portion of the block and comparing it to the radius of the sphere. In cases where the top of the sphere has been machined to accommodate a fastener, an allowance for the missing portion must be made. Verification of this requirement should be made prior to placing a block into service and whenever the bearing surface has been refinished. Due to the complex nature of measurements required, this verification should be made by a machine shop or an individual equipped with the tools to make these measurements. Although no tolerance is assigned to this requirement in the standard, it should be noted that after careful examination and review of research data, the upper spherical bearing block used for compression testing of concrete cylindrical specimens has been assigned a 5 % tolerance for this requirement.

FLOW TABLE

(See Specification C 230)²

47. Flow Table

47.1 The flow table top should be maintained in good condition. Avoid defacing the top with metal hand implements. The top must be clean, dry, and free of grease or oil when a flow test is made. In addition to maintaining the top in good condition, the table top must be level. This task can be accomplished by placing 4-in. square metal shims of various thickness between the cork pads and the floor. The leveling

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FIG. 3 Disassembled Upper Spherically Seated Bearing Block Displaying the Spherical Portion and Socket Portion of the Bearing Block that Requires Periodic Cleaning and Lubrication

should be checked at several points of reference on the table top. Due to possible compression of the cork pads, leveling should be checked periodically and adjusted, if necessary. Replacement of the cork pads at this time is advisable.

47.2 Prior to the start of daily testing, the table should be operated a dozen or more times. The vertical shaft should be cleaned frequently and reoiled with a light oil. Oil or dirt should not be present between the contact faces of the table top and frame. Oil on the cam face will lessen wear and promote smoothness of operation. It is important to keep the reducing gears of the motor drive mechanism packed with suitable grease.

47.3 The flow mold should rest evenly on the table top. A kitchen spoon or similar tool is useful in transferring mortar from the mixing bowl to the flow mold.

47.4 Specification C 230 states that the performance of a flow table is to be judged by the results obtained from flow tests made using a suitable calibration material. If the results of these tests are considered to be unsatisfactory, a thorough examination of the apparatus should be made. Some other important points to be considered during this examination are as follows:

47.4.1 Flow tables that do not conform to the general design requirements of C 230 are unreliable.

47.4.2 A pedestal must contain normal concrete in good condition. Pedestals, when new, should be moist-cured 28 days before use.

47.4.3 Flow tables located on flexible floors, such as old wooden floors, frequently fail to give satisfactory service. External vibrations may cause detrimental effects to the flow test.

47.4.4 Excessive rotation of the table top is undesirable. This problem may be caused by wear on the end of the vertical shaft.

47.4.5 The cam should meet the shaft smoothly, and the shaft should drop without hitting the cam.

47.4.6 The accuracy of the flow caliper, the weight of the table top, the height of drop, the rate of drop, and the planeness of the contact surfaces have an influence on test results.

47.4.7 Clearances between shaft and bore must be within specified limits both at the top and bottom.

47.4.8 The use of shims between table base and plate, or plate and pedestal, is undesirable.

47.4.9 Full contact between the metal plate and the pedestal is necessary. The plate should not overhang the pedestal and there should be no visible spaces between them.

47.4.10 Full contact between table base and metal plate is necessary. Check for the following:

47.4.10.1 Oil, rust, dirt, paint, etc., in the joint between the base and the plate,

47.4.10.2 Planeness of bottom surface of table base and top surface of plate, and

47.4.10.3 Nonplaneness in vicinity of tapped holes in the metal plate.

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47.4.11 Unseen cracks and voids in the table and pedestal can be detected with new technologies such as ultrasonic measurements.

AIR CONTENT OF MORTAR

(See Test Method C 185)²

48. Equipment

48.1 The measure should be kept scrupulously clean since accumulations of hardened cement or mortar on the inside of the measure will change the calibrated volume and tare weight leading to erroneous results. Accurate calibration is essential since any error will be reflected directly in the determinations of air content. Care should be used in handling the measure to avoid dents or other damage that will change the volume. Some measures are two piece, having a bottom that is press fit or soldered into the measure. This bottom should be checked periodically for a secure fit since any movement will change the volume of the measure.

49. Preparation and Testing of the Mortar

49.1 Because of the nature of this test, the accurate batching of all materials and the cleanliness and grading uniformity of the standard sand are of great importance in producing accurate results. This is also true with respect to the mixing procedure, flow determination, and the procedure for filling and weighing the measure. Close adherence to the timing and temperature requirements of the method are of extreme importance for the same reasons. It is because of the importance of each of these operations that the procedure for each is described in more explicit detail in the test method than is normal for some other less critical test methods. Departures from the stated procedures will give inaccurate and noncomparable results.

50. Causes of Variations Attributable to Test Procedure

50.1 *Use of an Incorrect Amount of Cement, Sand, or Water*—If water requirements for all cements seem to be consistently in error, the flow table and mechanical mixer should be thoroughly checked for conformance with the requirements of applicable specifications.

50.2 *Failure to Observe Specified Time Limits on Test Operations*—The desired accuracy cannot be obtained if the time limits on various test operations are disregarded.

50.3 *Failure to Use Specified Equipment in Performance of Test*—The specified spoon, tamper, tapping stick, and straightedge should be visually inspected periodically. Special attention should be given to the tapping stick as deterioration of the wood can change the effectiveness of the tapping.

50.4 *Failure to Place Mortar Gently in, or Overfill, the Flow Mold and 400-mL Measure*—The technique of throwing mortar into either the mold or measure does not conform with standard practice. Throwing mortar into the measure may cause entrapment of air voids in the mortar which can result in erroneous air contents. Pay particular attention to properly over filling the flow mold and 400-mL measure consistently by approximately 20 mm, as specified in the Test Method.

50.5 *Improper Handling of the 400-mL Measure*—Shaking or jolting of the measure during the filling operation must be avoided.

50.6 *Variations in Strike-Off Procedure*—The use of too few sawing strokes with the straightedge results in a tearing of the top surface of the mortar in the measure and the use of too many results in the formation of a ridged surface. A total of 10 to 12 strokes in each direction is recommended. Care should be taken to avoid trapping grains of sand between the measure and the straightedge.

51. Air Entraining Potential of Testing Sands

51.1 Difficulties have been experienced with Test Method C 185 in some laboratories due to the variable air entraining potential of the testing sands. These difficulties have usually taken the form of values for air content that are abnormally high and may be greater than the specified maxima in specifications for hydraulic cements. The air entraining potential of the testing sands may be reduced by washing by decantation with potable water until a clear supernatant liquid is obtained and then drying the sand before using in the test procedure.

STANDARD SAND

(See Test Method C 778)²

52. Gradation

52.1 *20–30 Sand*—A natural silica sand from Ottawa, IL or Lesueur, MN, used primarily in testing for the percentage of entrained air in hydraulic cements.

52.2 *Graded Sand*—A natural silica sand from Ottawa, IL used primarily in testing hydraulic cements for strengths.

52.3 A number of other types of sand are prepared by the processing plants. The laboratory is required to check each new shipment to ascertain that sand conforming to the requirements of the cement testing sand for 20–30 sands or graded sands has been received.

CALIBRATION AND CARE OF CEMENT TESTING EQUIPMENT

53. Calibration and Care of Apparatus

53.1 It is important that the apparatus used in cement testing be kept in good condition at all times. Apparatus should be inspected at frequent intervals to see that it is functioning properly and that it conforms to the requirements of the standard method involved. In such inspections, the laboratory should refer to the related paragraphs in this manual, and also to the latest applicable ASTM standard test methods. The good appearance of laboratory equipment is conducive to good work and therefore should not be neglected. Practice C 1222 provides some guidance on calibration or verification frequencies of equipment or both, as well as required documentation for those methods that do not specify a frequency.

CEMENT AND CONCRETE REFERENCE LABORATORY

54. Cement and Concrete Reference Laboratory

54.1 Since 1929, there has been maintained at the National Institute of Standards and Technology a Research Associate

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Program currently known as the Cement and Concrete Reference Laboratory. The CCRL is a cooperative project of the government and the American Society for Testing and Materials, sponsored by ASTM Committees C01 on Cement and C09 on Concrete and Concrete Aggregates. Its primary activities are inspection of laboratories and distribution of comparative test samples. At the request of the supervisor of a cement testing laboratory, the CCRL will schedule an on-site inspection of the procedures and equipment used by the laboratory in performing the standard physical tests for hydraulic cement, or register the laboratory for participation in a cement testing proficiency sample program covering the physical or chemical tests, or both for portland, masonry, and blended hydraulic cements. Charges for these services are made in accordance with fee schedules established by the sponsors. Inquiries should be addressed to the Cement and Concrete Reference Laboratory, National Institute of Standards and Technology, 100 Bureau Drive—Stop 8618, Gaithersburg, MD 20899-8618.

54.2 In addition to this service, it is suggested that each laboratory check its own apparatus and methods at regular intervals. Correspondence relative to standard test methods and apparatus will be welcomed by the Cement and Concrete Reference Laboratory.

TRAINING OF CEMENT TESTERS

55. Outline of Instruction

55.1 Considerable experience is necessary before an operator is able to secure consistent results in repeated tests on the same cement sample. Therefore, before assuming the responsibility of making tests which involve the acceptance of cement, the operator should be thoroughly trained in all details of the standard tests. The following outline of instruction is suggested:

55.1.1 Study thoroughly the various ASTM cement specifications and test methods, and also this manual. Training videos on various cement tests are available from the Portland Cement Association, Skokie, Illinois. Before using any training aide it should be thoroughly checked for agreement with current ASTM specifications and test methods.

55.1.2 Make normal consistency tests on several samples from the same lot of cement (at least 20 samples) until in successive tests the water requirements agree within 0.5 %. This will give practice in mixing and in preparing the sample for the determination of time of setting by the Vicat method. The normal consistency test is very important, for the quantity of mixing water used in some of the other tests depends on this determination.

55.1.3 Practice making pats until test specimens of the specified dimensions can be secured with a minimum of effort. Not less than 20 to 30 trials should be made. The top surface of Gillmore pats should be horizontal.

55.1.4 Make five test specimens each for time-of-setting tests by the Vicat and the Gillmore methods and determine the setting time by each method. Between intervals consumed in making observations of time of setting, the operator may continue his practice on other tests.

55.1.5 Make Blaine air-permeability fineness tests on several samples of each of a number of cements of different finenesses until the results of duplicate determinations on any given cement, made on separate beds, agree well within 2 % of each other.

55.1.6 Make Wagner turbidimeter fineness tests on several samples of each of a number of cements of different finenesses. Reference to some of the preceding comparative work in his own laboratory should readily give an operator a good idea of the proficiency which he has attained.

55.1.7 Make compressive strength tests for three or four ages (1, 3, 7, and 28 days) on each of at least ten samples from the same lot of cement. These mixes should be performed over several different days.

55.1.8 Autoclave tests should be made on five different days of at least three samples from the same lot of cement.

55.1.9 Make several air content determinations on a sample of non-air-entrained and on a sample of air-entrained cement, varying the water-to-cement ratio to produce flows within the range from 80 to 95 %.

55.1.10 Having attained a reasonable proficiency in a given test, the new operator should make parallel determinations with a skilled operator on five different days on five samples of cement. The samples should be from different lots of cement, preferably including different brands and types. Comparison between the skilled operator's results and his own will furnish the new operator with a good measure of his ability to make the various tests. Attention is again called to the advantages to be gained by regular, frequent interchange of samples for test by different laboratories.

55.1.11 Make early stiffening (paste method) tests on several samples from the same lot of cement (at least 20 samples) until in successive tests the water requirements agree with 0.5 % for initial penetration. Returning the material to the mixer and filling the ring mold several times can aid in reducing the time required to fill, strike off the mold, and release the plunger within 20 s.

55.1.12 Make No. 325 sieve fineness tests on several samples of each of a number of cements of different finenesses until the results of duplicate determinations on any given cement, made with different sieve screens, agree within the precision and bias statement.

SAFETY

56. Safety in the Cement Testing Laboratory

56.1 Contact with dry cement or freshly mixed (unhardened) cementitious material mixtures can cause skin irritation, severe chemical burns, or serious eye damage. Avoid contact with eyes and skin. Wear waterproof gloves, a fully buttoned long-sleeved shirt, full-length trousers, and eye protection such as safety glasses or safety goggles when working with these materials. Wash cement or freshly mixed (unhardened) cementitious material mixtures from your skin with fresh, clean water immediately after contact. Indirect contact through clothing can be as serious as direct contact, so promptly rinse out cement or freshly mixed

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(unhardened) cementitious material mixtures from clothing. Seek immediate medical attention if you have persistent or severe discomfort. In case of eye contact, flush with plenty of water for at least 15 min. Consult physician immediately.

56.2 In all cases the proper OSHA, MSHA, state, and local safety regulations must be followed. These are often minimum requirements in some areas, and should not prevent the cement testing laboratory from using initiative and resourcefulness in developing safer working environments. Examples of key safety equipment are safety showers, eye wash stations, and necessary personal protection equipment.

56.3 Safety must be one of the first considerations when designing a laboratory, training technicians, and in the daily operation of the laboratory. A modern laboratory often utilizes intricate combinations of energy, materials, and apparatus. Hazardous chemicals, radioactive materials, X-ray equipment, high voltage equipment, and compressed gases are commonly found in the cement laboratory. Reliable equipment and prime operating techniques are imperative for the safe and efficient performance of laboratory duties.

56.4 Testing facilities should be inspected to be certain that proper work areas and environment conditions are adequate for existing equipment. New apparatus should be installed in the laboratory only after necessary modifications have been made to the laboratory to accommodate the demands of the new equipment. A systematic maintenance and safety inspection system should be developed and utilized in each testing facility. The maintenance and operating instructions provided by the equipment manufacturer and current operating procedures should be readily available for each item of equipment. A record of repairs, adjustments, and service should be kept for each piece of apparatus.

56.5 Good safety practices must be insisted upon in every laboratory. Safety must be included in training programs for new personnel. Some of the ASTM test methods include safety requirements as part of the test method. These instructions and requirements should be rigidly followed.

56.6 The laboratory is often looked upon as a resource center for providing technical information on the handling, storage, and use of certain chemicals and materials that require special attention. Resource information such as MSDS on materials likely to be found in the facility must be readily available. A list of agencies and individuals who could provide specific and precise information on hazardous materials and conditions must be on hand for emergencies. Most laboratories and testing facilities are members of societies and trade associations such as the Portland Cement Association for cement producer member companies who have safety departments and established safety programs. The cement testing laboratory should avail themselves of these resource centers and facilities.

56.7 A bibliography of books and publications that have been found to be useful in the cement testing laboratory is provided at the end of this manual as a guide for selecting reference materials on laboratory safety.

56.8 The standards described in this manual may involve the use of hazardous materials, operations, and equipment. It is

the responsibility of whomever uses these standards to establish appropriate safety practices and to determine the applicability of regulatory limitations prior to use.

56.9 Proper storage of chemicals is essential to the safe functioning of the laboratory. For example, acids and bases should not be stored in the same cabinet. Furthermore, some acids should not be stored together (that is, oxidizing and organic). Consult the MSDS or other such reference for safe chemical storage. Flammable chemicals should be stored in a flammable/chemical cabinet. Chemicals need to be properly labeled and stored in a suitable container.

CHEMICAL ANALYSIS OF HYDRAULIC CEMENT

(See Test Methods C 114)²

57. Chemical Analysis

57.1 Method of Analysis:

57.1.1 The scope of Test Methods C 114 says, in essence, that any method may be used for analysis of hydraulic cement provided that it is demonstrated that the method, as used by the individual analyst, achieves required levels of precision and bias. The required way to accomplish that demonstration is spelled out in the section on *Qualification for Different Analyses* of Test Methods C 114. It requires qualification testing of the analyst for all test methods. When Rapid Test Methods are used, the test method must also be qualified. All qualifications must be made with Certified Reference Materials (CRM). Acceptable CRM materials are described in Section 4.1 of Test Methods C 114.

57.2 *Reasons for Qualification*—According to the requirements of the *Qualification for Different Analyses* section, qualification testing is required for any test method being used for any of the following reasons:

57.2.1 As referee analysis when there is a question about conformance to chemical specification requirements.

57.2.2 As analysis for acceptance or rejection of a cement.

57.2.3 As analysis for manufacturer's certification.

The amount of qualification testing required varies depending on the test method being used.

57.3 *Analyst Qualification*—Qualification testing must be performed on each test method (analyte) using one CRM when performing tests for any of the three reasons stated in the *Reasons for Qualification* section listed above. The test must be performed in duplicate with each test being performed on different days. An analyst is considered to be qualified for a test method when the testing of the CRMs does not vary by more than the limits set forth in Columns 2 and 3 of Table 1 of Test Methods C 114. Requalification of the analyst must be performed every two years.

57.4 *Test Method Qualification*—When performing tests for any of the three reasons stated in the *Reasons for Qualification* section, qualification testing is required for any test method not listed in the Reference Method Sections of Test Methods C 114. According to Test Methods C 114, at least seven CRMs must be used in the qualification. Each of the CRMs must be tested in duplicate on different days. To qualify a test method, the variation in the test results for six of the seven CRMs must be within the limits set forth in Column 3 of

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Table 1 of Test Methods C 114 and the remaining result must not be more than two times the limit shown in Column 3. If more than seven CRMs are used in the qualification process, at least 77 % of the results must be within the limits shown in Column 3 of Table 1 and the remaining results must be within two times the limit shown in Column 3 for the analyte being qualified. Requalification of a test method is required when there is "substantial evidence" that the test method is not providing results within the limits set forth in Table 1. Examples of "substantial evidence" are provided in 4.4.6.2 of Test Methods C 114. If the test methods used require qualification, one of the seven CRMs qualifying procedures may be used to qualify the analyst at the same time.

57.5 Information in the *General* Section of Test Methods C 114 applies to all methods. It provides general information on precautions, interferences and limitations, apparatus and

materials, reagents, and sample preparation. Routine procedures such as calculation and rounding of figures, and the recommended order of reporting analysis are found in this section.

57.6 The procedures starting in the *Reference Test Methods* Section and succeeding sections of Test Methods C 114 are, as noted in the Scope, not required for analysis of cements. They are in the books solely to serve as a guide for those who desire to use them. They are the classical wet chemical methods for analysis of cement proven over many years to be capable of providing precise and accurate results when used with proper care. They require no expensive instruments and can be run successfully by almost any normally equipped chemical laboratory. Practice and experience with these methods is very important since many of the procedures are technique dependent.

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