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## Standard Test Method for Time of Setting of Hydraulic Cement by Vicat Needle<sup>1</sup>

This standard is issued under the fixed designation C 191; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon ( $\epsilon$ ) indicates an editorial change since the last revision or reapproval.

### 1. Scope

1.1 These methods determine the time of setting of hydraulic cement by means of the Vicat needle. Two test methods are given; Method A is the Reference Test Method using the manually operated standard Vicat apparatus, while Method B permits the use of an automatic Vicat machine that has, in accordance with the qualification requirements of this method, demonstrated acceptable performance.

NOTE 1—For the method for determining time of setting by Gillmore needles, see Test Method C 266.

1.2 The values stated in SI units are to be regarded as the standard. Values in parentheses are for information only.

1.3 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.* See 1.4 for a specific warning statement.

1.4 **Warning:** Fresh hydraulic cementitious mixtures are caustic and may cause chemical burns to skin and tissue upon prolonged exposure. The use of gloves, protective clothing, and eye protection is recommended. Wash contact area with copious amounts of water after contact. Wash eyes for a minimum of 15 min. Avoid exposure of the body to clothing saturated with the liquid phase of the unhardened material. Remove contaminated clothing immediately after exposure.

### 2. Referenced Documents

#### 2.1 ASTM Standards:

- C 150 Specification for Portland Cement<sup>2</sup>
- C 187 Test Method for Normal Consistency of Hydraulic Cement<sup>2</sup>
- C 266 Test Method for Time of Setting of Hydraulic-Cement Paste by Gillmore Needles<sup>2</sup>
- C 305 Practice for Mechanical Mixing of Hydraulic Cement Pastes and Mortars of Plastic Consistency<sup>2</sup>
- C 490 Practice for Use of Apparatus for the Determination

of Length Change of Hardened Cement Paste, Mortar, and Concrete<sup>2</sup>

C 595 Specification for Blended Hydraulic Cements<sup>2</sup>

C 1005 Specification for Reference Masses and Devices for Determining Mass and Volume for Use in the Physical Testing of Hydraulic Cements<sup>2</sup>

C 1157 Performance Specification for Blended Hydraulic Cement<sup>2</sup>

### 3. Apparatus

3.1 *Reference Masses and Devices for Determining Mass*, conforming to the requirements of Specification C 1005. The devices for determining mass shall be evaluated for precision and bias at a total load of 1 kg.

3.2 *Glass Graduates*, 200 or 250-mL capacity, and conforming to the requirements of Specification C 490.

### 4. Temperature and Humidity

4.1 The temperature of the air in the vicinity of the mixing slab, the dry cement, molds, and base plates shall be maintained between 20 and 27.5°C (68 and 81.5°F). The temperature of the mixing water and of the moist closet or moist room shall not vary from 23°C (73.4°F) by more than  $\pm 1.7^\circ\text{C}$  (3°F).

4.2 The relative humidity of the laboratory shall be not less than 50%. The moist closet or moist room shall be so constructed as to provide storage facilities for test specimens at a relative humidity of not less than 90%.

### 5. Preparation of Cement Paste<sup>3</sup>

5.1 Mix 650 g of cement with the percentage of mixing water required for normal consistency following the procedure described in Practice C 305. Distilled water is preferable and shall be used for all referee or cooperative tests.

5.2 At the option of the tester, use the test specimen used for determining normal consistency, or mix a new batch for determining time of setting by Method A.

5.3 For Method B, use the paste remaining from the batch used for the normal consistency for molding the test specimen or mix a new batch (see Note 2).

NOTE 2—The specimen used for the determination of the normal consistency will have an irregular surface, making it unsuitable for the automatic setting time apparatus.

<sup>1</sup> This method is under the jurisdiction of ASTM Committee C01 on Cement and is the direct responsibility of Subcommittee C01.30 on Time of Set.

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<sup>2</sup> *Annual Book of ASTM Standards*, Vol 04.01.

<sup>3</sup> See Test Method C 187.

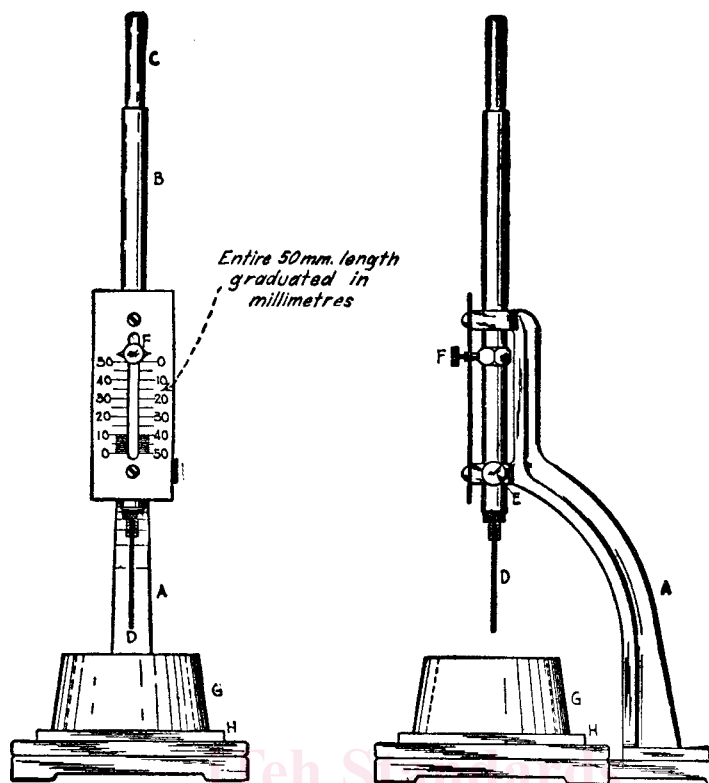


FIG. 1 Vicat Apparatus

#### Method A—Manual Vicat Needle Apparatus

### 6. Manual Vicat Apparatus

6.1 The Vicat apparatus shall consist of a frame, A, Fig. 1, bearing a movable rod, B, weighing 300 g, one end, C, the plunger end, being 10 mm in diameter for a distance of at least 50 mm and the other end having a removable steel needle, D, 1 mm in diameter and 50 mm in length. The rod B is reversible, and can be held in any desired position by a set screw, E, and has an adjustable indicator, F, which moves over a scale (graduated in millimetres) attached to the frame, A. The paste is held in a conical ring, G, resting on a plate of similar planeness, corrosivity, and absorptivity to that of glass, H, about 100 mm square. The ring shall be made of a noncorroding, nonabsorbing material, and shall have an inside diameter of 70 mm at the base and 60 mm at the top and a height of 40 mm. In addition to the above, the Vicat apparatus shall conform to the following requirements:

Weight of plunger	300 ± 0.5 g (0.661 lb ± 8 grains)
Diameter of larger end of plunger	10 ± 0.05 mm (0.394 ± 0.002 in.)
Diameter of needle	1 ± 0.05 mm (0.039 ± 0.002 in.)
Inside diameter of ring at bottom	70 ± 3 mm (2.75 ± 0.12 in.)
Inside diameter of ring at top	60 ± 3 mm (2.36 ± 0.12 in.)
Height of ring	40 ± 1 mm (1.57 ± 0.04 in.)
Graduated scale	The graduated scale, when compared with a scale accurate to within 0.1 mm at all points, shall not show any point greater than 0.25 mm

### 7. Procedure

7.1 *Molding Test Specimen*—Quickly form the cement paste, prepared as described in the section on preparation of

cement paste, into a ball with the gloved hands and toss six times from one hand to the other, maintaining the hands about 6 in. (152 mm) apart. Press the ball, resting in the palm of the hand, into the larger end of the conical ring, G, Fig. 1, held in the other hand, completely filling the ring with paste. Remove the excess at the larger end by a single movement of the palm of the hand. Place the ring on its larger end on a plate of similar planeness, corrosivity, and absorptivity to that of glass, H, and slice off the excess paste at the smaller end at the top of the ring by a single oblique stroke of a sharp, straight-edged trowel held at a slight angle with the top of the ring. Smooth the top of the specimen, if necessary, with one or two light touches of the pointed end of the trowel. During the operation of cutting and smoothing, take care not to compress the paste. Immediately after molding, place the test specimen in the moist closet or moist room and allow it to remain there except when determinations of time of setting are being made. The specimen shall remain in the conical mold, supported by the plate of similar planeness, corrosivity, and absorptivity to that of glass, H, throughout the test period. A time of setting specimen and an autoclave bar may be made from the same batch.

7.2 *Time of Setting Determination*— Allow the time of setting specimen to remain in the moist cabinet for 30 min after molding without being disturbed. Determine the penetration of the 1-mm needle at this time and every 15 min thereafter (every 10 min for Type III cements) until a penetration of 25 mm or less is obtained. For the penetration test, lower the needle D of the rod B until it rests on the surface of the cement paste. Tighten the set screw, E, and set the indicator, F, at the upper end of the scale, or take an initial reading. Release the rod quickly by releasing the set screw, E, and allow the needle to

settle for 30 s; then take the reading to determine the penetration. (If the paste is obviously quite soft on the early readings, the fall of the rod may be retarded to avoid bending the 1-mm needle, but the rod shall be released only by the set screw when actual determinations for the time of setting are made.) Make each penetration test at least 5 mm away from any previous penetration and at least 10 mm away from the inner side of the mold. Record the results of all penetration tests and, by interpolation, determine the time when a penetration of 25 mm is obtained. This is the initial setting time. The final setting time is when the needle does not sink visibly into the paste.

**7.3 Precautions**—All the apparatus shall be free from vibration during the penetration test. Keep the 1-mm needle straight and clean. The needle must be kept clean because cement adhering to the sides of the needle may retard penetration, while cement on the point may increase the penetration. The time of setting is affected not only by the percentage and the temperature of the water used and the amount of kneading the paste received, but also by the temperature and humidity of the air, and its determination is therefore only approximate.

## 8. Precision and Bias

### 8.1 Precision:

**8.1.1** The single-operator (within-laboratory) standard deviation has been found to be 12 min for the initial time of setting, throughout the range of 49 to 202 min, and 20 min for the final time of settings throughout the range of 185 to 312 min. Therefore, results of two properly conducted tests by the same operator on Vicat initial time of setting of similar paste should not differ from each other by more than 34 min and on Vicat final time of setting of similar pastes should not differ from each other by more than 56 min.

**8.1.2** The multilaboratory (between-laboratory) standard deviation has been found to be 16 min for the initial time of setting throughout the range of 49 to 207 min, and 43 min for the final time of setting throughout the range of 185 to 312 min. Therefore, results of two properly conducted tests from two different laboratories on Vicat initial time of setting of similar pastes should not differ from each other by more than 45 min, and on Vicat final time of setting of similar pastes should not differ from each other by more than 122 min.

**8.2 Bias**—Since there are no accepted reference materials suitable for determining the bias for the procedure in this test method, no statement on bias is presented.

## Method B—Automatic Vicat Needle Apparatus

### 9. Automatic Vicat Apparatus

**9.1 Automatic Vicat Needle Apparatus**—The apparatus shall be equipped essentially with a standard Vicat needle. The needle shall have a  $1.0 \pm 0.05$  mm diameter and have a length of at least 50 mm. The total mass supported by the needle tip at the time of measurement shall be  $300 \pm 0.5$  g. The instrument shall be capable of automatically completing and recording penetration measurements of a test specimen at predetermined time intervals not exceeding 10 min and make each penetration test at least 5 mm away from any previous penetration and at least 10 mm away from the inner side of the mold.

**9.2 Specimen Mold**—The cement paste is held in a conical ring with the height of  $40 \pm 1$  mm and a removable base plate. The test surface shall have a minimum diameter of  $60 \pm 3$  mm.

## 10. Procedure

**10.1 Molding the Specimen**—Quickly form the cement paste, prepared as described in the section on preparation of cement paste, into a ball with the gloved hands and toss six times from one hand to the other, maintaining the hands about 150 mm apart. Press the ball, resting in the palm of the hand, into the larger end of the conical ring, held in the other hand, completely filling the ring with paste. Remove the excess at the larger end by a single movement of the palm of the hand. Place the ring on its larger end on the base plate. Use a sharp, straight-edged trowel to remove the excess paste at the top of the mold. Holding the trowel at about a  $30^\circ$  incline with the leading edge raised and starting near the center of the mold, level the paste by drawing the trowel across the top of the mold using a sawing motion; repeat the procedure for the other half of the surface. Then smooth the surface level to the top of the mold making steady and complete strokes across the entire surface with the trailing edge of the trowel. Repeat the cutting and smoothing steps but at  $90^\circ$  from the previous cut. Repeat the steps as required to produce a surface level with the top of the mold. Usually the paste is level within two cycles, but occasionally three will be required. Avoid excessive strokes and compaction. Since the automatic Vicat device references to the top edge of the mold, it is imperative that the top surface of the paste be uniform and level with the top of the mold.

**10.2 Time of Setting Determinations**—Follow the manufacturer's instructions and complete the required instrument calibration and zero procedures. Set the instrument to measure no less frequently than every 10 min (see Note 3). Position the molded specimen in the automatic Vicat apparatus and initiate the measurements (see Note 4).

**NOTE 3**—Automatic devices are usually capable of measurement more frequently than once every 10 min and more frequent measurement minimizes interpolation.

**NOTE 4**—The initial time of setting according to Method A is determined as the elapsed time required to achieve a penetration of 25 mm and the final setting as the total elapsed time when the needle does not sink visibly into the paste. The end points established by a qualified automatic Vicat test method may deviate significantly from end points specified in Method A, and require standardization to correct bias.

**10.3** The range of applicability of the method shall be determined by the range of the average time of setting of the cements used for qualification and standardization, and shall be limited to 30 min greater than the maximum, and 30 min less than the minimum.

**10.4 Calculations and Reporting**—Complete all calculations to the nearest 1 min, and report results to the nearest 5 min. The report shall indicate that tests were conducted using Method B.

## 11. Performance Requirement (Qualification) for Automatic Vicat Instrument

**11.1 Scope**—When time of setting results from the automatic Vicat apparatus are to be used for acceptance or rejection of cement, the method used shall comply with the qualification