



## **Standard Test Methods 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 reappraisal. A superscript epsilon ( $\epsilon$ ) indicates an editorial change since the last revision or reappraisal.

### **1. Scope**

1.1 These test 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.

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.<sup>2</sup>

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

### **2. Referenced Documents**

#### *2.1 ASTM Standards:*<sup>3</sup>

C 150 Specification for Portland Cement

C 151 Test Method for Autoclave Expansion of Portland Cement

C 183 Practice for Sampling and the Amount of Testing of Hydraulic Cement

C 187 Test Method for Normal Consistency of Hydraulic Cement

C 219 Terminology Relating to Hydraulic Cement

C 266 Test Method for Time of Setting of Hydraulic-Cement Paste by Gillmore Needles

C 305 Practice for Mechanical Mixing of Hydraulic Cement Pastes and Mortars of Plastic Consistency

C 511 Specification for Mixing Rooms, Moist Cabinets, Moist Rooms, and Water Storage Tanks Used in the Testing of Hydraulic Cements and Concretes

C 595 Specification for Blended Hydraulic Cements

C 1005 Specification for Reference Masses and Devices for Determining Mass and Volume for Use in the Physical Testing of Hydraulic Cements

C 1157 Performance Specification for Blended Hydraulic Cement

D 1193 Specification for Reagent Water

### **3. Terminology**

3.1 *Definitions*—The terms used in this test method are defined in accordance with Terminology C 219.

### **4. Summary of Test Method**

4.1 A paste that is proportioned and mixed to normal consistency, as described in the Test Method C 187, is molded and placed in a moist cabinet and allowed to start setting. Periodic penetration tests are performed on this paste by allowing a 1-mm Vicat needle to settle into this paste. The Vicat initial time of setting is calculated as the time elapsed between the initial contact of cement and water and the time when the penetration is at 25 mm. The Vicat final time of setting is calculated as the time elapsed between initial contact of cement and water and the time when the needle does not sink visibly into the paste.

### **5. Significance and Use**

5.1 This test method provides a means of determining compliance with a specification limit for Vicat time of setting. Refer to the appropriate specification for the cement to determine if this test method is used for specification compliance.

<sup>1</sup> These test methods are 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> Section on Safety, Manual of Cement Testing, *Annual Book of ASTM Standards*, Vol 04.01.

<sup>3</sup> 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.

5.2 The measured time of setting is affected by the percentage and temperature of the water used, the amount of kneading the paste received, and by the temperature and humidity of the mixing room air and the moist cabinet or moist room air.

5.3 The measured time of setting of hydraulic cement is test-method specific. Time of setting as measured by this method will not necessarily be similar to other methods used for determining the time of setting of hydraulic cements.

## 6. Apparatus

6.1 *Vicat Apparatus*—See Annex A1.1 and Fig. A1.1. The Vicat apparatus for this test method shall have a movable rod, B, of mass  $300 \pm 0.5$ g. The end of the rod used for measuring penetration shall have a removable needle, D, with a diameter of  $1.00 \pm 0.05$  mm and length no less than 50 mm.

6.2 *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.

6.3 *Glass Graduates*, 200 or 250-mL capacity, and conforming to the requirements of Specification C 1005.

6.4 *Plane non-adsorptive plate*,  $100 \pm 5$  mm square of similar planeness, corrosivity, and absorptivity to that of glass (see Annex A1.1, Fig. A1.1, H).

6.5 *Flat trowel*, having a sharpened straight-edged steel blade 100 to 150 mm in length.

6.6 *Conical ring*, made of a rigid, non-corroding, non-absorbent material and shall have a height of  $40 \pm 1$  mm, an inside diameter at the bottom of  $70 \pm 3$  mm, and an inside diameter at the top of  $60 \pm 3$  mm (see Annex A1.1, Fig. A1.1, G).

6.7 *Mixer, bowl, and paddle*, conforming to Practice C 305.

6.8 *Automatic Vicat Needle Apparatus for Method B*—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.

6.9 *Specimen Mold for Method B*—The cement paste is held in a conical ring as described in 6.6.

## 7. Reagents and Materials

7.1 *Mixing Water*—Potable water is satisfactory for routine tests. Use water conforming to the requirements of Specification D 1193 for Type III or Type IV grade reagent water for all referee and cooperative tests.

## 8. Sampling

8.1 When the test is required for acceptance testing, sample cement in accordance with Practice C 183.

## 9. Conditioning

9.1 Maintain the temperature of the air in the vicinity of the mixing slab, the dry cement, molds, and base plates at  $23.0 \pm 3.0^\circ\text{C}$ .

9.2 Maintain the temperature of the mixing water at  $23.0 \pm 2.0^\circ\text{C}$ .

9.3 The relative humidity of the mixing room shall be not less than 50 %.

9.4 The moist cabinet or moist room shall be in accordance with Specification C 511.

## 10. Preparation of Cement Paste<sup>4</sup>

10.1 The cement paste used for the determination of the time of setting is obtained from one of the following methods:

10.1.1 Prepare a new batch of paste by mixing 650 g of cement with the percentage of mixing water required for normal consistency (Test Method C 187), following the procedure described in Practice C 305.

10.1.2 For method A, at the option of the tester, use the test specimen used for determining normal consistency (see Note 2).

10.1.3 At the option of the tester, use the paste remaining from the batch used for the autoclave specimen (Test Method C 151) or from the normal consistency determination (Test Method C 187).

NOTE 2—The specimen used for the determination of the normal consistency will have an irregular surface, making it unsuitable for method B.

## 11. Calculation

11.1 Calculate the Vicat time of setting to the nearest 1 min as follows:

$$\left( \left( \frac{H-E}{C-D} \right) \times (C-25) \right) + E \quad (1)$$

where:

*E* = time in minutes of last penetration greater than 25 mm,

*H* = time in minutes of first penetration less than 25 mm,

*C* = penetration reading at time *E*, and

*D* = penetration reading at time *H*.

11.2 Calculate the Vicat final time of setting by determining the elapsed time between the time of the initial contact between cement and water and the time when the needle does not sink visibly into the paste, rounded to the nearest 5 min.

## 12. Report

12.1 Report the time of setting and the method used as follows:

Vicat time of setting (A or B) \_\_\_\_\_ min  
 Vicat final time of setting (A or B) \_\_\_\_\_ min

### Method A—Manual Vicat Needle Apparatus

## 13. Manual Vicat Apparatus

13.1 The Vicat apparatus shall consist of a frame, A, Fig. A1.1, bearing a movable rod, B, weighing 300 g, one end, C,

<sup>4</sup> See Test Method C 187.

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

#### 14. Procedure A

14.1 *Molding Test Specimen*—Quickly form the cement paste, prepared as described in the section on preparation of cement paste, into a ball with gloved hands and toss six times from one hand to the other, maintaining the hands about 150 mm (6 in.) apart. Press the ball, resting in the palm of the hand, into the larger end of the conical ring, *G*, Fig. A1.1, 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 onto the non-absorptive plate, *H*, and slice off the excess paste at the smaller end at the top of the ring by a single oblique stroke of the 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 cabinet or moist room and allow it to remain there except when penetration measurements are being made. The specimen shall remain in the conical mold, supported by the non-absorptive plate throughout the test period.

14.2 *Time of Setting Determination*—Allow the time of setting specimen to remain in the moist cabinet or moist room 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. Perform the penetration test by lowering the needle *D* of the rod *B* until it rests on the surface of the cement paste. Tighten the setscrew, *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. At the option of the tester, if the paste is obviously quite soft on the early readings, retard the fall of the rod to avoid bending the 1-mm needle, but when

actual penetration measurements to determine the time of setting are made, release the rod only by the setscrew. 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. The elapsed time between the initial contact of cement and water and the penetration of 25 mm is the Vicat time of setting or Vicat initial time of setting.

14.3 Determine the Vicat final time of setting end point to be the first penetration measurement that does not mark the specimen surface with a complete circular impression, verified by two additional penetration measurements performed on different areas of the specimen surface. The elapsed time between the initial contact of cement and water and the end point determination above is the Vicat final time of setting.

14.4 *Precautions*—Keep all the apparatus free from vibration during the penetration test. Keep the 1-mm needle straight and clean. The needle must be kept clean to prevent cement from adhering to the sides of the needle and decreasing penetration, and to prevent cement from adhering to the point and increasing penetration.

#### 15. Precision and Bias

##### 15.1 Precision:

15.1.1 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.

15.1.2 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.

15.2 *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

#### 16. Automatic Vicat Apparatus

16.1 *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.

16.2 *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.

## 17. Procedure

17.1 *Molding the Specimen*—Quickly form the cement paste, prepared as described in the section on preparation of cement paste, into a ball with 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 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.

17.2 *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 time of setting as the total time elapsed until 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.

17.3 Determine the range of applicability of the method from the range of the average time of setting of the cements used for qualification and standardization, and limit that range to 30 min greater than the maximum, and 30 min less than the minimum.

17.4 *Performance Requirement (Qualification) for Automatic Vicat Instrument:*

17.4.1 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 requirements covered in Annex A2.

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

18.1 *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 requirements of this section. A method is considered to consist of the specific instrument and the molding procedure meeting the requirements of this standard, and used in a consistent manner by a given laboratory.

18.2 *Hydraulic Cement Samples Required*—Select three cements that comply with requirements of Specifications C 150, C 595, or C 1157, and that when tested in accordance with Method A, shall include the following:

18.2.1 One cement that has an initial time of setting of less than 110 min,

18.2.2 One cement with an initial time of setting of greater than 150 min,

18.2.3 One cement with a final time of setting of less than 180 min, and

18.2.4 One cement with a final time of setting of greater than 210 min (see Note 5).

NOTE 5—Laboratories are advised to reserve sufficient cement for future qualification and standardization.

18.3 *Tests*—Using the method to be qualified and including the standardization formula described in the section on standardization, make single determinations of the times of initial and final setting on each of the three cements. On the same day, conduct companion time of setting determinations according to Method A. Make single determinations of the normal consistency on each cement, and use that amount of water for all replicate batches. Complete three rounds of tests on different days, repeating all the steps of the methods. Conduct the qualification tests on specimens prepared separately from the standardization testing.

18.4 *Calculations*—Calculate the three-round averages for initial and final time of setting for each cement and method. A method complies with the initial time of setting qualification requirements if the difference between the initial time of setting average values for Method A and the corresponding average values of Method B is not greater than 25 min for any sample, and the range for any three replicate tests by Method B does not exceed 30 min. Likewise, a method complies with the final time of setting qualification requirements if the difference between the final time of setting average values for Method A and corresponding average values for Method B is not greater than 45 min for any sample, and the range for any three replicate tests by Method B does not exceed 30 min. Example qualification data are given in Appendix X1.

18.5 *Standardization*—When standardization is required in order to achieve agreement between Method A and Method B, it can be applied to either the initial time of setting, final time of setting, or both. A standardization formula shall be based on a comparison of test results obtained using Method A and the chosen method using the automatic instrument (Method B). Results of tests from at least five different hydraulic cements shall be required for standardization. The cements shall have a minimum range of 60 min initial time of setting, and a minimum range of 90 min final time of setting when tested in