Designation: C451 – 13

Standard Test Method for Early Stiffening of Hydraulic Cement (Paste Method)[1]

This standard is issued under the fixed designation C451; 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 (ε) indicates an editorial change since the last revision or reapproval.

1. Scope*

1.1 This test method covers the determination of early stiffening in hydraulic-cement paste.

1.2 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

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.*

Warning: Fresh hydraulic-cementitious mixtures are caustic and may cause chemical burns to skin and tissue upon prolonged exposure.[2]

1.4 The text of this standard references notes and footnotes which provide explanatory material. These notes and footnotes (excluding those in tables and figures) shall not be considered as requirements of the standard.

2. Referenced Documents

2.1 *ASTM Standards:*[3]

C150 Specification for Portland Cement

C183 Practice for Sampling and the Amount of Testing of Hydraulic Cement

C187 Test Method for Amount of Water Required for Normal Consistency of Hydraulic Cement Paste

C219 Terminology Relating to Hydraulic Cement

C305 Practice for Mechanical Mixing of Hydraulic Cement Pastes and Mortars of Plastic Consistency

C670 Practice for Preparing Precision and Bias Statements for Test Methods for Construction Materials

C1005 Specification for Reference Masses and Devices for Determining Mass and Volume for Use in the Physical Testing of Hydraulic Cements

D1193 Specification for Reagent Water

3. Terminology

3.1 *Definitions:*

3.1.1 *early stiffening, n*—the early development of stiffness in the working characteristics of a hydraulic-cement paste, mortar, or concrete; varieties include false set and flash set.

3.1.2 *false set, n*—the early development of stiffness in the working characteristics of a hydraulic-cement paste, mortar, or concrete without the evolution of much heat, which stiffness can be dispelled and plasticity regained by further mixing without addition of water; also known as “grab set,” “premature stiffening,” “ hesitation set,” and “rubber set.”

3.1.3 *flash set, n*—the early development of stiffness in the working characteristics of a hydraulic-cement paste, mortar, or concrete, usually with the evolution of considerable heat, which stiffness cannot be dispelled nor can the plasticity be regained by further mixing without addition of water; also known as “quick set.”

3.1.4 Refer to Terminology C219 for definitions of other terms.

4. Summary of Test Method

4.1 A paste is prepared with the cement to be tested, using sufficient water to give a required initial penetration as measured by the Vicat apparatus at a stipulated time after completion of mixing. A second penetration, termed the final penetration, is measured at a later stipulated time. The ratio of final penetration to initial penetration is calculated as a percentage.

5. Significance and Use

5.1 The purpose of this test method is to determine the degree to which a cement paste develops early stiffening or to establish whether or not a cement complies with a specification limit on early stiffening.

5.2 When used for establishing compliance with a specification limit, the specification requirement is customarily stated in terms of the minimum allowable final penetration, in percent, calculated in accordance with the Calculation Section.

[1] This test method is under the jurisdiction of ASTM Committee C01 on Cement and is the direct responsibility of Subcommittee C01.30 on Time of Set.

Current edition approved Dec. 1, 2013. Published January 2014.. Originally approved in 1960. Last previous edition approved in 2008 as C451 – 08. DOI: 10.1520/C0451-13.

[2] Section on Safety, Manual of Cement Testing, *Annual Book of ASTM Standards*, Vol 04.01.

[3] 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.

***A Summary of Changes section appears at the end of this standard**

When used for estimating the relative tendency of a cement to manifest early stiffening, additional information of value may be obtained if the remix procedure described in the Procedure Section is employed. Under some conditions, a judgment may be made by comparing the behavior in the initial test and in the remix procedure to differentiate a relatively less serious and less persistent tendency to early stiffening (false set) from one that is more persistent and, consequently, more serious (flash set).

5.3 Severe false setting in a cement may cause difficulty from a placing and handling standpoint, but it is not likely to cause difficulties where concrete is mixed for a longer time than usual, as usually occurs in transit mixing, or where it is remixed prior to placing or transporting, as occurs in concrete pumping operations. It is most likely to be noticeable where concrete is mixed for a short period of time in stationary mixers and transported to the forms in non-agitating equipment, such as on some paving jobs, and when concrete is made in an on-site batch plant.

5.4 Cements with severe false setting usually require slightly more water to produce the same consistency, which may be expected to result in slightly lower strengths and increased drying shrinkage.

5.5 Early stiffening resulting from false set is not likely to cause a cement to fail the applicable time of setting requirement.

5.6 Early stiffening resulting from flash set, depending on severity, can cause a cement to fail the applicable time of setting requirement.

6. Apparatus

6.1 *Vicat Apparatus,* conforming to the requirements of Test Method C187.

6.2 *Flat Trowel,* having a sharpened straight-edged steel blade 100 to 150 mm in length. The edges when placed on a plane surface shall not depart from straightness by more than 1 mm.

6.3 *Mixer, Bowl, Paddle, and Scraper,* conforming to the requirements of Practice C305.

6.4 *Glass Graduates,* 200 or 250 ml capacity, conforming to the requirements of Specification C1005.

6.5 *Masses and Mass Determining Devices,* conforming to the requirements of Specification C1005. The devices for determining mass shall be evaluated for precision and accuracy at a total load of 1000 g.

6.6 *Conical Ring,* made of a rigid, non-corroding, non-absorbernt material, having a height of 40 ± 1 mm, an inside diameter at the bottom of 70 ± 3 mm, and an inside diameter at the top of 60 ± 3 mm (see Test Method C187, Fig. 1 item G).

6.7 *Plane Non-Absorptive Plate,* 100 ± 5 mm square, of similar planeness, corrosivity, and absorptivity to that of glass (see Test Method C187, Fig. 1 item H).

6.8 Inspect and document Section 6 apparatus conformance to the requirements of this test method at least every 2 ½ years.

7. Reagent

7.1 *Mixing Water*—Potable water is satisfactory for routine tests. For all referee and cooperative tests, use reagent water conforming to the requirements of Specification D1193 for Type III or Type IV grades of reagent water.

8. Sampling

8.1 When the test is part of acceptance testing, sample the cement in accordance with Practice C183.

9. Conditioning

9.1 Maintain the temperature of the room, dry materials, paddle, bowl, conical ring, and base plate at 23.0 ± 3.0 °C. Maintain the temperature of the mixing water at 23.0 ± 2.0 °C.

9.2 Maintain the relative humidity of the mixing room at not less than 50 %.

10. Procedure

10.1 *Preparation of Cement Paste*—Mix 500 g of cement with sufficient water to produce a paste with an initial penetration of 32 ± 4 mm using the following procedure.

10.1.1 Place the dry paddle and the dry bowl in the mixing position in the mixer.

10.1.2 Introduce the materials for a batch into the bowl and mix in the following manner:

10.1.2.1 Place all the mixing water in the bowl.

10.1.2.2 Add the cement to the water and allow 30 s for the water to absorb.

10.1.2.3 Start the mixer and mix at a slow speed (140± 5 r/min) for 30 s.

10.1.2.4 Stop the mixer for 15 s, and during this time scrape down into the batch any paste that may have collected on the sides of the bowl.

10.1.2.5 Start the mixer at a medium speed (285 ±10 r/min) and mix for 2½ min.

10.2 *Molding Test Specimens*—Quickly form the cement paste into a ball with gloved hands. Press the ball, resting in the palm of one 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 non-absorptive plate and slice off the excess paste at the smaller end at the top of the ring by a single oblique stroke of a sharp-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.

10.3 *Determination of Initial Penetration*—Set the paste confined in the ring resting on the plate, H, under the rod, B, Fig. 1 of Test Method C187, about one third of the diameter from the edge, and bring the plunger end, C, in contact with the surface of the paste and the tightened setscrew, E. Then set the movable indicator, F, to the upper zero mark of the scale, and release the rod exactly 20 s after completion of the mixing. Keep the apparatus free of all vibrations during the penetration test. Consider the paste to have proper consistency when the

rod settles to a point 32 ± 4 mm below the original surface in 30 s after being released. Make trial pastes with varying percentages of water until this consistency is obtained. This consistency is the initial penetration. During the 30-s interval for the initial penetration, return the excess paste to the bowl and cover the bowl and mixing paddle with a lid.

10.4 *Determination of Final Penetration*—After completion of the initial reading, remove the plunger from the paste, clean it, and reset the ring and plate in a new position. Perform this operation with as little disturbance as possible to the paste confined in the Vicat ring. Then bring the plunger again in contact with the surface of the paste, tighten the setscrew, and set the movable indicator to the upper zero mark of the scale. Release the plunger a second time 5 min ± 10 s after completion of the mixing period, and determine the final penetration 30 s after the plunger is released.

10.5 *Determination of Remix Penetration*—If the penetrations determined by the foregoing procedure show the cement to be stiffening rapidly, at the option of the tester, obtain information as to the nature of the stiffening by testing as follows:

10.5.1 After completing the measurement of the 5-min penetration, immediately return the paste in the ring to the bowl.

10.5.2 Start the mixer, raise the bowl into mixing position, and remix the contents of the bowl at a medium speed (285 ±10 r/min) for 1 min.

10.5.3 Fill the ring and determine the penetration following the procedures specified in 10.2 and 10.3.

11. Calculation

11.1 Calculate the percent final penetration, based on the ratio of final penetration to initial penetration, as follows:

$$P, \% = \frac{B}{A} \times 100 \quad (1)$$

where:

P = percent final penetration,
A = initial penetration, mm, and
B = final penetration, mm.

12. Report

12.1 Report the results of the test as follows:

Initial penetration	___ mm
Final penetration	___ mm
Percent final penetration	___ %
Remix penetration	___ mm

13. Precision and Bias

13.1 Precision

13.1.1 On samples testing between 8 % and 89 % Paste False Set, the single-operator (within laboratory) standard deviation has been found to be 10 % (1s), therefore, results of two properly conducted tests by the same operator on samples of the same cement should not differ from each other by more than 28 % (1s and d2s are defined in Practice C670).

13.1.2 On samples testing between 8 % and 89 % Paste False Set, the multi-laboratory standard deviation has been found to be 12 % (1s), therefore, results of two properly conducted tests from two different laboratories on samples of the same cement should not differ by more than 34 % (1s and d2s are defined in Practice C670). (See Note 1.)

13.2 Bias

13.2.1 Since an acceptable reference material suitable for determining any bias of this method does not exist, no statement on bias is being made.

NOTE 1—This data is based upon CCRL Portland Cement round robin test samples 47 to 138. The data indicates that precision decreases as the false set values fall below approximately 60 %. These decreases are not clearly statistically significant, however they do indicate the duplication difficulties on cements which exhibit false set tendencies severe enough to fail or possibly fail Specification C150 optional limits.

14. Keywords

14.1 early stiffening; false set; flash set; hydraulic-cement paste; Vicat

SUMMARY OF CHANGES

Committee C01 has identified the location of selected changes to this test method since the last issue, C451 – 08, that may impact the use of this test method. (Approved Dec. 1, 2013)

(1) Revised warning statement in 1.3.