



Designation: C 1608 – 06

Standard Test Method for Chemical Shrinkage of Hydraulic Cement Paste¹

This standard is issued under the fixed designation C 1608; 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 approval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

1. Scope*

1.1 This test method measures the internal (absolute) volume change of hydraulic cement paste that results from the hydration of the cementitious materials. This volume change is known as *chemical shrinkage*.

1.1.1 Procedure A, volumetric method.

1.1.2 Procedure B, the density method.

1.2 The values stated in SI units are to be regarded as the 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*

2. Referenced Documents

2.1 *ASTM Standards*:³

C 186 Test Method for Heat of Hydration of Hydraulic Cement

C 188 Test Method for Density of Hydraulic Cement

C 219 Terminology Relating to Hydraulic Cement

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

3. Terminology

3.1 *Definitions*:

¹ This test method is under the jurisdiction of ASTM Committee C01 on Cement and is the direct responsibility of Subcommittee C01.31 on Volume Change.

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² See the 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.

3.1.1 *chemical shrinkage, n*—the absolute (internal) volume change accompanying the hydration of cement, due to the fact that the cement hydration products occupy less physical volume than the reactants.

3.1.2 All other terms are as defined in Terminology C 219.

4. Significance and Use

Numerous properties of cementitious materials are controlled by their initial hydration rate. Examples include early-age strength development, heat release, and crack resistance. One direct and convenient measure of this initial hydration rate is provided by the measurement of the chemical shrinkage of the cement paste during its hydration. As cement hydrates, the hydration products occupy less volume than the initial reacting materials (cement and water). Due to this volume change, a hydrating cement paste will sorb water from its immediate surroundings, when available. At early times, this sorption is in direct proportion to the amount of hydration that has occurred.⁴ This method is based on the one developed by Geiker.⁵ The results are relevant to understanding the hydration behavior of cements. This method does not measure the bulk volume changes (autogenous shrinkage) associated with chemical shrinkage nor the cracking potential of concretes produced with the evaluated cement.

5. Apparatus

5.1 *Devices for Determining Mass*, conforming to the requirements of Specification C 1005 and evaluated for precision and accuracy at a total load of 100 g.

5.2 *Constant Temperature Water Bath*—a water bath capable of maintaining a temperature of 23.0 ± 0.5 °C, with a sufficient capacity to hold the specimens being evaluated. To avoid evaporative cooling, the surface of the water in the bath shall be covered with floating plastic balls or fitted with an insulated lid.

5.3 *Timing Device*—Clock that can measure time to the nearest minute.

⁴ L.J. Parrott, M. Geiker, W.A. Gutteridge, and D. Killoh, "Monitoring Portland Cement Hydration: Comparison of Methods," *Cement and Concrete Research*, Vol. 20, 919-926, 1990.

⁵ M. Geiker, "Studies of Portland Cement Hydration: Measurements of Chemical Shrinkage and a Systematic Evaluation of Hydration Curves by Means of the Dispersion Model," Ph.D. Thesis, Technical University of Denmark, Copenhagen, Denmark, 1983.

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

5.4 For procedure A

5.4.1 *Capillary Tube*—A graduated glass capillary tube with graduations of 0.01 mL or smaller, and typically a capacity of 1.0 mL.

5.4.2 *Small Glass Vials (e.g., 22-mm diameter and 55-mm height) with Rubber Stoppers* that fit tightly into the glass vials and have a hole placed in each stopper with the graduated capillary tube inserted through the hole (as shown in Fig. 1). Fix the capillary tube in the stopper using a two-component epoxy or other suitable adhesive applied at the stopper's top and bottom surfaces.

5.5 For Procedure B

5.5.1 *Density bottle*, glass, capacity approximately 20 ml with internally conical glass stopper as shown in Fig. 2.

6. Reagents and Materials

6.1 Paraffin oil.

6.2 De-aerated water (prepared by boiling water and sealing it in a closed container before it has cooled.)

7. Procedure

7.1 *Preparation of Cement Paste*—Prepare the cement paste in accordance with the proportions and procedure described in Test Method C 186 (Note 1). The pastes shall be prepared in a mixing room meeting the temperature and humidity requirements outlined in Specification C 511. Record to the nearest minute the time when the water first contacts the dry cement powder.

NOTE 1—Other mixing procedures such as mixing in a Hobart mixer (see Practice C 305) or kneading by hand in a sealed plastic bag may be used. The standard paste mixture uses 150 g of cement and 60 ml of water (water-cement ratio of 0.40). Adjust the volume of paste mixed to match the mixing equipment used. Other water-cement ratios may be used as

long as they are stated in the test report; higher water-cement ratios may produce significant bleeding of the cement paste which will influence the results (by changing the effective water-cement ratio, etc.); lower water-cement ratios may lead to difficulties in preparing a fully compacted, homogeneous paste for subsequent evaluation and self-desiccation may occur.

7.2 Prepare a minimum of two replicate specimens as described below for either Procedure A or Procedure B.

7.3 Procedure A

7.3.1 Determine the mass of each empty glass vial to the nearest 0.01 g.

7.3.2 Carefully place the prepared cement paste into the glass vial to achieve a paste height between 5 mm and 10 mm in the vial. Consolidate the paste in the vial by tapping the vial on a laboratory countertop, or placing it on a vibrating table, or some similar procedure.

7.3.3 Determine the mass of each glass vial with the cement paste to the nearest 0.01g.

7.3.4 Carefully, without disturbing the cement paste, add clean, de-aerated water to fill the glass vial to the top.

7.3.5 Place the rubber stopper with the inserted capillary tube tightly into the glass vial. Be careful to avoid the entrapment of air bubbles when the bottom rubber stopper surface encounters the water in the glass vial. As the rubber stopper is inserted, the water level in the graduated capillary tube will rise. Optimally, the water level should rise near to but not beyond the top mark of the graduations on the capillary tube. If the water level is not near enough to the top mark, clean, de-aerated water can be added via the top of the capillary tube to achieve the desired initial water height. Once the rubber stopper has been fitted to the vial, the completed assembly shall be handled only by the vial (not by the capillary tube) to avoid

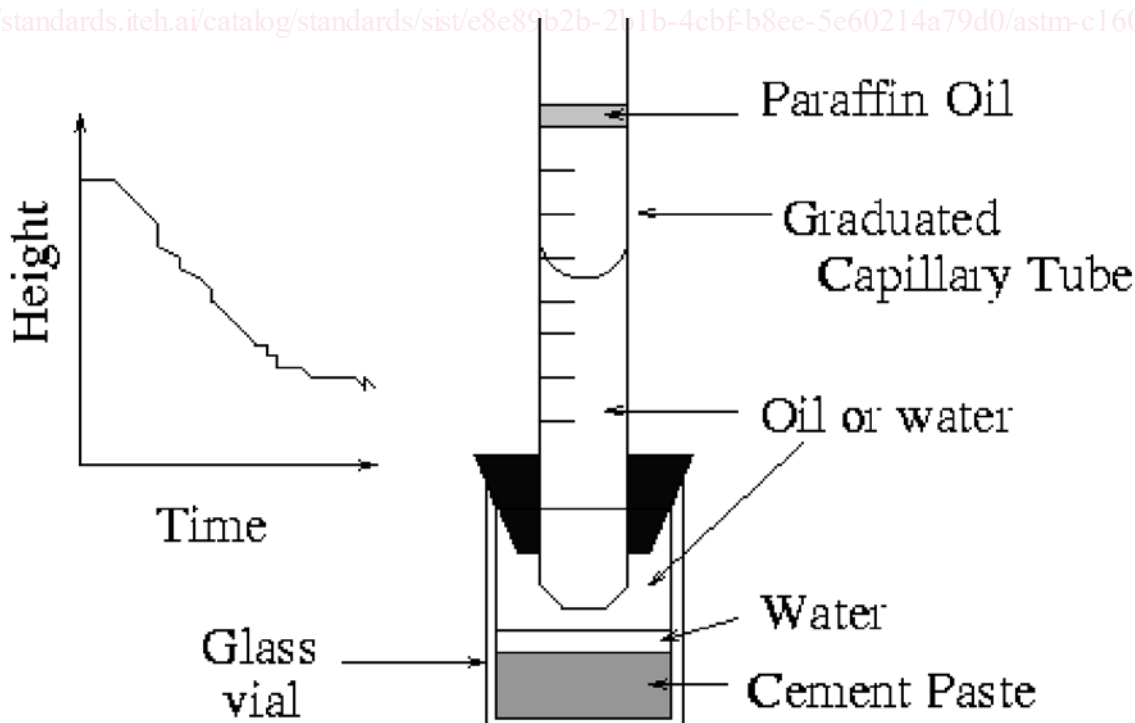


FIG. 1 Illustration of One Experimental Setup for Monitoring Chemical Shrinkage of Hydrating Cement Paste Using Procedure A.