



Designation: C1012/C1012M – 13

# Standard Test Method for Length Change of Hydraulic-Cement Mortars Exposed to a Sulfate Solution<sup>1</sup>

This standard is issued under the fixed designation C1012/C1012M; 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 This test method covers the determination of length change of mortar bars immersed in a sulfate solution. Mortar bars made using mortar described in Test Method C109/C109M are cured until they attain a compressive strength of  $20.0 \pm 1.0$  MPa [ $3000 \pm 150$  psi], as measured using cubes made of the same mortar, before the bars are immersed.

1.2 The values stated in either SI units or inch-pound units are to be regarded separately as standard. Within the text, the inch-pound units are shown in brackets. The values stated in each system may not be exact equivalents; therefore, each system shall be used independently of the other. Combining values from the two systems may result in non-conformance with 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.*

## 2. Referenced Documents

### 2.1 ASTM Standards:<sup>2</sup>

- C109/C109M Test Method for Compressive Strength of Hydraulic Cement Mortars (Using 2-in. or [50-mm] Cube Specimens)
- C114 Test Methods for Chemical Analysis of Hydraulic Cement
- C150 Specification for Portland Cement
- C157/C157M Test Method for Length Change of Hardened Hydraulic-Cement Mortar and Concrete
- C215 Test Method for Fundamental Transverse, Longitudinal, and Torsional Resonant Frequencies of Concrete Specimens

- C305 Practice for Mechanical Mixing of Hydraulic Cement Pastes and Mortars of Plastic Consistency
- C348 Test Method for Flexural Strength of Hydraulic-Cement Mortars
- C349 Test Method for Compressive Strength of Hydraulic-Cement Mortars (Using Portions of Prisms Broken in Flexure)
- C452 Test Method for Potential Expansion of Portland-Cement Mortars Exposed to Sulfate
- C490 Practice for Use of Apparatus for the Determination of Length Change of Hardened Cement Paste, Mortar, and Concrete
- C511 Specification for Mixing Rooms, Moist Cabinets, Moist Rooms, and Water Storage Tanks Used in the Testing of Hydraulic Cements and Concretes
- C595 Specification for Blended Hydraulic Cements
- C597 Test Method for Pulse Velocity Through Concrete
- C618 Specification for Coal Fly Ash and Raw or Calcined Natural Pozzolan for Use in Concrete
- C684 Test Method for Making, Accelerated Curing, and Testing Concrete Compression Test Specimens (Withdrawn 2012)<sup>3</sup>
- C778 Specification for Sand
- C917 Test Method for Evaluation of Cement Strength Uniformity From a Single Source
- C989 Specification for Slag Cement for Use in Concrete and Mortars
- D1193 Specification for Reagent Water
- E18 Test Methods for Rockwell Hardness of Metallic Materials
- E177 Practice for Use of the Terms Precision and Bias in ASTM Test Methods
- E691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method

### 2.2 American Concrete Institute Reports: ACI C201-2R-01 Guide to Durable Concrete<sup>4</sup>

<sup>1</sup> This test method is under the jurisdiction of ASTM Committee C01 on Cement and is the direct responsibility of Subcommittee C01.29 on Sulfate Resistance.

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<sup>2</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.

<sup>3</sup> The last approved version of this historical standard is referenced on www.astm.org.

<sup>4</sup> Available from American Concrete Institute (ACI), P.O. Box 9094, Farmington Hills, MI 48333-9094, http://www.concrete.org.

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

### 3. Significance and Use

3.1 This test method provides a means of assessing the sulfate resistance of mortars made using portland cement, blends of portland cement with pozzolans or slags, and blended hydraulic cements. Method C452 is suitable for evaluating portland cements but not blended cements or blends of portland cement with pozzolans or slags.

3.2 The standard exposure solution used in this test method, unless otherwise directed, contains 352 moles of  $\text{Na}_2\text{SO}_4$  per  $\text{m}^3$  (50 g/L). Other sulfate concentrations or other sulfates such as  $\text{MgSO}_4$  may be used to simulate the environmental exposure of interest. Further discussion of these and other technical issues is given in the Appendix.

### 4. Apparatus

4.1 *Mixer*, conforming to the requirements of Practice C305.

4.2 *Cube Molds*, conforming to the requirements of Test Method C109/C109M.

4.3 *Bar Molds*, conforming to the requirements of Specification C490.

4.4 *Comparator*, conforming to the requirements of Specification C490.

4.5 *Containers*—The containers in which the bars are immersed in the sulfate solution shall be corrosion resistant such as plastic, glass, or ceramic. Support the bars so that no end or side of a bar rests against the container. Seal the container with a lid so that the sulfate solution cannot evaporate.

4.6 *Initial Curing Container*—The container and the risers in which the specimens are to be cured shall be corrosion and heat resistant such as plastic, glass, or ceramic. The container shall be of a size that permits the storage of the specimens, so that no end or side of a bar or cube rests against the container. The lid on the container shall be air tight, so that the water cannot evaporate. More than one container may be required for the initial curing of a complete set of bars and cube specimens.

4.7 *Oven*—A convection oven with temperature control maintaining  $35 \pm 3^\circ\text{C}$  ( $95 \pm 5^\circ\text{F}$ ).

### 5. Reagents and Materials

5.1 *Purity of Reagents*—USP or technical grade chemicals may be used, provided it is established that any reagent used is of sufficiently high purity to permit its use without lessening the accuracy of the determination. When tests are made that are expected to produce results that are close to an acceptance-rejection value, it is recommended that reagent grade chemicals be used. Such chemicals shall conform to the specifications of the Committee on Analytical Reagents for the American Chemical Society where such specifications are available.<sup>5</sup>

<sup>5</sup> *Reagent Chemicals, American Chemical Society Specifications*, American Chemical Society, Washington, DC. For Suggestions on the testing of reagents not listed by the American Chemical Society, see *Annual Standards for Laboratory Chemicals*, BDH Ltd., Poole, Dorset, U.K., and the *United States Pharmacopeia and National Formulary*, U.S. Pharmacopeial Convention, Inc. (USPC), Rockville, MD.

5.2 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean reagent water conforming to Type IV of Specification D1193.

5.3 *Sodium Sulfate* ( $\text{Na}_2\text{SO}_4$ )—Check the water content by loss on ignition each time the solution is prepared. Any anhydrous or hydrated sodium sulfate may be used if the water content of the salt is checked by loss on ignition and proper corrections made to account for the specified sulfate concentration.

5.4 *Sulfate Solution*—Each litre of solution shall contain 50.0 g of  $\text{Na}_2\text{SO}_4$  dissolved in 900 mL of water, and shall be diluted with additional distilled or deionized water to obtain 1.0 L of solution. Mix the solution on the day before use, cover, and store at  $23.0 \pm 2.0^\circ\text{C}$  [ $73.5 \pm 3.5^\circ\text{F}$ ]. Determine the pH of the solution before use; reject the solution if the pH range is outside 6.0 to 8.0. Maintain the volume proportion of sulfate solution to mortar bars in a storage container at  $4.0 \pm 0.5$  volumes of solution to 1 volume of mortar bars. For mortar bars 1 by 1 by 11¼ in. (volume of 184 mL [ $11.25 \text{ in.}^3$ ]), this is 645 to 830 mL of solution per mortar bar in the storage container. For mortar bars 25 by 25 by 285 mm (volume 178 mL), this is 625 to 800 mL of solution per mortar bar in the storage container.

5.5 *Materials*:

5.5.1 *Graded Standard Sand*, as specified in Specification C778.

5.5.2 *Stainless Steel Gauge Studs*, as specified in Specification C490.

### 6. Hazards

6.1 **Warning**—Fresh hydraulic cementitious mixtures are caustic and may cause chemical burns to skin and tissue upon prolonged exposure.<sup>6</sup>

### 7. Preparing Mortars

7.1 Make mortars as described in Test Method C109/C109M, that is, 1 part cement to 2.75 parts of sand by mass. Use a water-cement ratio by mass of 0.485 for all non-air-entraining portland cements and 0.460 for all air-entraining portland cements. Use a water-cement ratio by mass of 0.485 for non-air-entraining portland-pozzolan (IP) and portland-blast furnace slag (IS) cements. For blends of portland cement with a pozzolan or slag, use a water-cement ratio that develops a flow within  $\pm 5$  of that of the portland-cement mortar at a water-cement ratio of 0.485.

### 8. Specimen Molds and Initial Curing Container

8.1 Prepare the specimen molds in accordance with the requirements of Specification C490 except the interior surfaces of the mold shall be covered with a release agent. A release agent will be acceptable if it serves as a parting agent without affecting the setting of the cement and without leaving any residue that will inhibit the penetration of water into the specimen.

<sup>6</sup> See *Manual of Cement Testing*, Section on Safety, *Annual Book of ASTM Standards*, Vol 04.01.

NOTE 1—TFE-fluorocarbon tape complies with the requirements for a mold release agent.

## 8.2 Prepare the Curing Container:

8.2.1 Prior to molding the test specimen, prepare the initial curing container by placing risers on the bottom of the container to elevate molds above the intended water level. Fill the container with preheated water at  $35 \pm 3^\circ\text{C}$  ( $95 \pm 5^\circ\text{F}$ ), making sure that the water does not exceed the top of the risers.

## 9. Procedure

9.1 *Molding and Initial Curing of Specimens*—Mold the test bars in accordance with Test Method **C157/C157M**. Mold the cubes in accordance with Test Method **C109/C109M**. A set of specimens to test one cementitious product consists of 6 bars and up to 21 cubes (**Note 2**). Immediately after molding cover the molds with a rigid steel, glass, or plastic plate, and place the mold in the curing container on top of the risers. Cover the container with a lid and seal the lid so as to prevent evaporation and place the container into an oven at  $35 \pm 3^\circ\text{C}$  ( $95 \pm 5^\circ\text{F}$ ) for  $2\frac{1}{2}$  h  $\pm$  30 min. At  $2\frac{1}{2}$  h  $\pm$  30 min, remove molds from container and demold the specimens.

NOTE 2—The set of cubes consists of 21 cubes to be tested as described herein when significant information on the strength development rate is not available. When information is available (as for example, from the use of the procedures of Test Method **C917**) that would justify making fewer cubes, only those needed to confirm the time the mortar achieves  $20.0 \pm 1.0$  MPa [ $3000 \pm 150$  psi] are needed.

9.2 *Subsequent Curing and Preparation for Test*—After demolding, store all bars and cubes, except the two to be tested, in a curing tank of saturated limewater at  $23.0 \pm 2.0^\circ\text{C}$  [ $73.5 \pm 3.5^\circ\text{F}$ ]. Test two cubes in compression in accordance with Test Method **C109/C109M** after demolding when the specimens have cooled to ambient temperature under moist cloths. If the mean strength of the two cubes is 20 MPa [2850 psi] or more, observe and record comparator readings in accordance with Specification **C490** and as prescribed in the section on Measurements of Length Change and place all the bars in the sulfate solution. If 20 MPa [2850 psi] is not achieved, store the demolded cubes and mortar bars in the curing tank and test additional cubes (see **Note 3**). Predict from the first two cubes when a compressive strength of at least 20 MPa [2850 psi] will be reached. Verify the prediction, and at that time observe and record comparator readings and place all the bars in the sulfate solution (**Note 3**). This measurement is designated as the initial length. The storage temperature and test temperature shall be  $23.0 \pm 2.0^\circ\text{C}$  [ $73.5 \pm 3.5^\circ\text{F}$ ].

NOTE 3—If the value for strength at 24 h is less than 20.0 MPa [2850 psi] and additional testing on the same day is not possible, or, is unlikely to yield a value over 20.0 MPa [2850 psi] and the strength is over 21 MPa [3150 psi] when tested early the next day, it is not necessary to remake the batch. If the two cubes do not meet the required strength, continue curing the cubes and bars as referenced in **9.2**, in a curing tank of saturated limewater at  $23.0 \pm 2.0^\circ\text{C}$  [ $73.5 \pm 3.5^\circ\text{F}$ ].

9.3 *Storage of Test Bars during Exposure to Test Solution*—Cover the container of the bars and test solution, and seal it to prevent evaporation from the inside, or dilution with water from the outside. (See **Note 4**.) The storage temperature and test temperature shall be  $23.0 \pm 2.0^\circ\text{C}$  [ $73.5 \pm 3.5^\circ\text{F}$ ]. (See **Note 5**.)

NOTE 4—Gaffers tape or duct tape has been found to be suitable for sealing the container.

NOTE 5—This is the same temperature and temperature range as that specified for moist rooms in Specification **C511**.

9.4 *Measurements of Length Change*—At 1, 2, 3, 4, 8, 13, and 15 weeks after the bars are placed in the sulfate solution, test them for length change using the length comparator in accordance with Specification **C490**. Review the data at 15 weeks. Make the subsequent measurements at 4, 6, 9, and 12 months. If at any time the rate of change between readings is great, insert other readings as needed to ensure the intervals are short enough to permit observing and reporting the behavior of the bars. If compliance with ACI C201-2R-01 Class 3 exposure is required, measure the bars at 4, 6, 9, 12, 15, and 18 months (**Note 6**).

NOTE 6—ACI C201-2R-01 requires an 18-month limit for Class 3 exposure.

### 9.4.1 Details of Measurement of Bars for Length Change:

9.4.1.1 Clean the hole in the base of the comparator into which the gauge stud on the lower end of the bar fits (this hole tends to collect water and sand and should be cleaned after every reading). Read and record the comparator indication of the length of the reference bar. Take one bar out of immersion, blot the pins, put the bar in the comparator, read, and record the indication. Return the bar to immersion and clean the hole in the base of the comparator. Take out the second bar and treat it in a like manner. Return the second bar to immersion, record the reading, and clean the hole in the base of the comparator. Continue the procedure until all bars have been read, returned to immersion, and the readings recorded, cleaning the hole in the bottom of the comparator each time. After reading the last bar, clean the hole in the comparator base and read and record the reference-bar indication.

9.4.1.2 When the required strength of the mortar is achieved in accordance with **9.2**, store the bars in fresh sulfate solution. At subsequent readings for length change, proceed as described in **9.4.1.1**; cleaning the socket in the base of the comparator before reading the reference bar initially and after reading each mortar bar. Record reference bar and mortar bar readings. Read and record the reference bar again after measurement of the last bar. Blot only around the pins (**Note 7**). Return each bar to the used sulfate solution after reading. Discard the used solution after reading of the bars at only standard intervals: 1, 2, 3, 4, 8, 13, and 15 weeks and 4, 6, 9, 12, 15, and 18 months. Rinse the container once with water, pouring out water and debris. Replace the frame holding the bars in the container, fill the container with enough new sulfate solution to immerse bars, and secure the lid on the container.

NOTE 7—The purpose of the minimal blotting of the pins and no blotting of the bars is to avoid drying and shrinkage of the bars. It has been observed that if the pins are blotted, and the bar placed in the comparator and the dial read, and the bar is then wiped gently with a dry cloth, the bar will shrink measurably. Therefore, drying should be minimized.

9.4.2 *Examination of Specimens After Measuring Length Change*—When the bars seem to have behaved in an unusual way or when the test is part of a research study, test the specimens for warping by placing them on a plane surface so that the ends are curved down and the maximum bowing

measured. Note cracking (presence, location, type); also note surface deposits, mottling, exudations (nature, thickness, type).

9.5 *Tolerance on Time*—All references to elapsed time in 9.4 are intended to have a tolerance of  $\pm 2\%$ .

**TABLE 1 Maximum Permissible Range of Values**

Remaining No. of Specimens	Blended Cements	Portland Cement
3	0.034	0.010
4	0.037	0.011
5	0.039	0.012
6	0.041	0.012

## 10. Calculation

10.1 Calculate the length change at any age as follows:

$$\Delta L = \frac{L_x - L_i}{L_g} \times 100 \quad (1)$$

where:

- $\Delta L$  = change in length at  $x$  age, %,
- $L_x$  = comparator reading of specimen at  $x$  age—reference bar comparator reading at  $x$  age, and
- $L_i$  = initial comparator reading of specimen—reference bar comparator reading, at the same time
- $L_g$  = nominal gauge length, or 250 mm [10 in.] as applicable. (See C490).

10.2 Calculate length change values for each bar to the nearest 0.001 % and report averages to the nearest 0.01 %.

## 11. Report

11.1 Report type of cement, and, if blending material is used, its identification and amount and whether cement and blending material, if used, meet the applicable specifications. Report the initial comparator reading of each bar at the time of immersion in sulfate solution; this is the base value for calculation of length changes. Report the subsequent length changes in percent of base value to the nearest 0.001 % for individual bars and the nearest 0.01 % for averages. Comment on the visual condition of bars at end of the test. With each report of mean length change of bars at a particular age, report number of bars ( $n$ ), standard deviation of length change, and coefficient of variation of length change. Data from at least three bars must be available at any age to constitute a valid test at that age. Depending upon how many remaining specimens there are, the maximum permissible range of the values must not exceed the values of length change in percent in Table 1.

11.2 Report any variation from the procedure outlined in the text as it relates to solution composition, concentration, or temperature, mortar proportions, age, or maturity, and so forth.

## 12. Precision and Bias

12.1 The precision of this test method has been evaluated by cooperative testing and found to vary with the type of cement studied, hence separate precision statements are given.<sup>7</sup>

12.1.1 *Blended Cements*—The single-operator standard deviation has been found to be 0.010 % for expansions between 0.04 % and 0.07 %. Therefore, results of two properly conducted tests by the same operator on the same blended cement Type IP or IS, should not differ from each other by more than 0.028 %. The multi-laboratory standard deviation has been found to be 0.020 % for expansions between 0.04 %, and 0.07 %. Therefore, results of two properly conducted tests on the same blended cement Type IP or IS in two different laboratories, should not differ from each other by more than 0.056 %.

12.1.2 *Type II Cement*—The single-operator standard deviation has been found to be 0.005 %, for expansions between 0.04 % and 0.07 %. Therefore, results of two properly conducted tests by the same operator on the same cement, should not differ from each other by more than 0.014 %. The multi-laboratory standard deviation has been found to be 0.020 % for expansions between 0.04 % and 0.07 %. Therefore, results of two properly conducted tests on the same cement in two different laboratories, should not differ from each other by more than 0.056 %.

12.1.3 *Type V Cement*—The single-operator standard deviation has been found to be 0.003 % for expansions between 0.04 % and 0.07 %. Therefore, results of two properly conducted tests by the same operator on the same cement, should not differ from each other by more than 0.009 %. The multi-laboratory standard deviation has been found to be 0.010 % for expansions between 0.04 % and 0.07 %. Therefore, results of two properly conducted tests on the same cement in two different laboratories should not differ from each other by more than 0.028 %.

12.2 *Bias*—Since there is no accepted reference material suitable for determining the bias for the procedure in this test method, no statement on bias is being made.

## 13. Keywords

13.1 acceptability; expansion; fly ash; mortars; pozzolans; precision; search report; slag; sulfate attack; sulfate-resisting cements; tests

<sup>7</sup> Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR: RR:C01-1003.