

Designation: C1012/C1012M - 24

Standard Test Method for Length Change of Hydraulic-Cement Mortars Exposed to a Sulfate Solution¹

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 (ε) 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 MPa \pm 1.0 MPa [3000 psi \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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.
- 1.4 This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.

2. Referenced Documents

2.1 ASTM Standards:²

C109/C109M Test Method for Compressive Strength of Hydraulic Cement Mortars (Using 50 mm [2 in.] Cube Specimens)

C114 Test Methods for Chemical Analysis of Hydraulic Cement

C150/C150M 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/C490M 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/C595M Specification for Blended Hydraulic Cements C597 Test Method for Ultrasonic Pulse Velocity Through Concrete

C618 Specification for Coal Ash and Raw or Calcined Natural Pozzolan for Use in Concrete

C778 Specification for Standard Sand

C917/C917M Test Method for Evaluation of Variability of Cement from a Single Source Based on Strength

C989/C989M Specification for Slag Cement for Use in Concrete and Mortars

C1157/C1157M Performance Specification for Hydraulic

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

 $^{^{\}rm 1}$ 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.

Current edition approved April 1, 2024. Published April 2024. Originally approved in 1984. Last previous edition approved in 2018 as C1012/C1012M – 18b. DOI: 10.1520/C1012_C1012M-24.

² 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.

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 Concrete³

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. Test 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 Na_2SO_4 per $m^3(50 \text{ g/L})$. Other sulfate concentrations or other sulfates such as $MgSO_4$ 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/C490M.
- 4.4 *Comparator*, conforming to the requirements of Specification C490/C490M.
- 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 °C \pm 3 °C (95 °F \pm 5 °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 specifica-

³ Available from American Concrete Institute (ACI), P.O. Box 9094, Farmington Hills, MI 48333-9094, http://www.concrete.org.

tions of the Committee on Analytical Reagents for the American Chemical Society where such specifications are available.⁴

- 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 (Na₂SO₄)—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 Na_2SO_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 °C ± 2.0 °C [73.5 °F ± 3.5 °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 ± 0.5 volumes of solution to 1 volume of mortar bars. For mortar bars 1 in. by 1 in. by $11 \frac{1}{4}$ in. (volume of 184 mL 11.25 in. 11.25
 - 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/C490M.

6. Hazards

6.1 **Warning**—Fresh hydraulic cementitious mixtures are caustic and may cause chemical burns to skin and tissue upon prolonged exposure.⁵

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 Specification C150/C150M cements and 0.460 for all air-entraining Specification C150/C150M cements. Use a water-cement ratio by mass of 0.485 for non-air-entraining Specification C595/C595M or C1157/C1157M cements. For mixtures consisting of a Specification C150/C150M, C595/C595M, or C1157/C1157M cement and a pozzolan or slag cement, use a water-cementitious materials ratio that develops

⁴ ACS Reagent Chemicals, Specifications and Procedures for Reagents and Standard-Grade Reference Materials, American Chemical Society, Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see Analar 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 See Manual of Cement Testing, Section on Safety, Annual Book of ASTM Standards, Vol 04.01.

a flow within ± 5 of that of the cement mortar without additional pozzolan or slag cement 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/C490M 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.

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

- 8.2 Prepare 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 °C \pm 3 °C (95 °F \pm 5 °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 °C \pm 3 °C (95 °F \pm 5 °F) for 23 ½ h \pm 30 min. At 23 ½ 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/C917M) that would justify making fewer cubes, only those needed to confirm the time the mortar achieves $20.0 \text{ MPa} \pm 1.0 \text{ MPa} [3000 \text{ psi} \pm 150 \text{ psi}]$ 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 °C ± 2.0 °C [73.5 °F \pm 3.5 °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/C490M 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 °C \pm 2.0 °C [73.5 °F \pm 3.5 °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 °C \pm 2.0 °C [73.5 °F \pm 3.5 °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 °C \pm 2.0 °C [73.5 °F \pm 3.5 °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 week, 2 weeks, 3 weeks, 4 weeks, 8 weeks, 13 weeks, 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/C490M. Review the data at 15 weeks. Make the subsequent measurements at 17 weeks, 26 weeks, 39 weeks, and 52 weeks. 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 the 18-month expansion limit for Exposure Class S3 is required, measure the bars at 17 weeks, 26 weeks, 39 weeks, 52 weeks, 65 weeks, and 78 weeks (Note 6).

Note 6—Some documents referencing this method include limits on expansion of mortars at 6 months, 12 months, or 18 months. These times are sufficiently close to 26 weeks, 52 weeks, and 78 weeks, respectively, within the 2 % tolerance on elapsed time in subsection 9.5, that mortar expansions at those times can be used to evaluate whether a mortar meets those limits. For example, ACI PRC 201.2-23 references an 18-month expansion limit for Exposure Class S3, and the same limit can be evaluated at 78 weeks.

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 week, 2 weeks, 3 weeks, 4 weeks, 8 weeks, 13 weeks, 15 weeks, 17 weeks, 26 weeks, 39 weeks, 52 weeks, 65 weeks, and 78 weeks. 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 ± 2 %.

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10. Calculation

10.1 Calculate the length change at any age as follows:

$$\Delta L = \frac{L_x - L_i}{L_g} \times 100 \qquad \underline{ASTM C10 (1)}$$
standards.iteh.ai/catalog/standards/astm/0e57b4a0-

where:

 ΔL = change in length at x age, %,

 L_x = comparator reading of specimen at x age—reference bar comparator reading at x age,

 L_i = initial comparator reading of specimen-reference bar comparator reading, at the same time, and

 L_g = nominal gauge length, or 250 mm [10 in.] as applicable (see Practice C490/C490M).

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. Include the length change of all measurable bars in the reported average. A valid length change determination shall be the average of at least three bars at each test age.

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.

Note 8—Guidance on preparing a test report is provided in Appendix

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.⁶

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 multilaboratory 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 multilaboratory 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

⁶ Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR: RR:C01-1003.