



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

This standard is issued under the fixed designation C 1012; 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 C 109/ C 109M are cured until they attain a compressive strength of 20.0 ± 1.0 MPa (3000 ± 150 psi), as measured using cubes made of the same mortar, before the bars are immersed.

1.2 The values stated in SI units are to be regarded as the standard. The values shown in parentheses are for information purposes 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.*

2. Referenced Documents

2.1 ASTM Standards:²

- C 109/C 109M Test Method for Compressive Strength of Hydraulic Cement Mortars (Using 2-in. or [50mm] Cube Specimens)
- C 114 Test Method for Chemical Analysis of Hydraulic Cement
- C 150 Specification for Portland Cement
- C 157/C 157M Test Method for Length Change of Hardened Hydraulic-Cement, Mortar, and Concrete
- C 215 Test Method for Fundamental Transverse, Longitudinal, and Torsional Frequencies of Concrete Specimens
- C 305 Practice for Mechanical Mixing of Hydraulic Cement Pastes and Mortars of Plastic Consistency
- C 348 Test Method for Flexural Strength of Hydraulic Cement Mortars
- C 349 Test Method for Compressive Strength of Hydraulic Cement Mortars (Using Portions of Prisms Broken in Flexure)

- C 452 Test Method for Potential Expansion of Portland-Cement Mortars Exposed to Sulfate
 - C 490 Practice for Use of Apparatus for the Determination of Length Change of Hardened Cement Paste, Mortar, and Concrete
 - 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 597 Test Method for Pulse Velocity Through Concrete
 - C 618 Specification for Coal Fly Ash and Raw or Calcined Natural Pozzolan for Use as a Mineral Admixture in Concrete
 - C 684 Test Method for Making, Accelerated Curing, and Testing Concrete Compression Test Specimens
 - C 778 Specification for Standard Sand
 - C 917 Test Method for Evaluation of Cement Strength Uniformity From a Single Source
 - C 989 Specification for Ground Granulated Blast-Furnace Slag for Use in Concrete and Mortars
 - D 1193 Specification for Reagent Water
 - E 18 Test Methods for Rockwell Hardness and Rockwell Superficial Hardness of Metallic Materials
- ### 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. Method C 452 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 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.

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.

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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.

³ Available from American Concrete Institute (ACI), P.O. Box 9094, Farmington Hills, MI 48333.

4. Apparatus

4.1 *Mixer*, conforming to the requirements of Practice C 305.

4.2 *Cube Molds*, conforming to the requirements of Test Method C 109/C 109M.

4.3 *Bar Molds*, conforming to the requirements of Specification C 490.

4.4 *Comparator*, conforming to the requirements of Specification C 490.

4.5 *Containers*—The containers in which the bars are immersed 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 *Curing Tank*, conforming to the requirements of Test Method C 684.

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.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 D 1193.

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 Na₂SO₄ 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 ± 2.0°C (73.5 ± 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 by 1 by 1 1/4 in. (volume of 184 mL or 11.25 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:

⁴ *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.

5.5.1 *Graded Standard Sand*, as specified in Specification C 778.

5.5.2 *Stainless Steel Gage Studs*, as specified in Specification C 490.

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 C 109/C 109M, 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 ±5 of that of the portland-cement mortar at a water-cement ratio of 0.485.

8. Specimen Molds

8.1 Prepare the specimen molds in accordance with the requirements of Specification C 490 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.

9. Procedure

9.1 *Molding and Initial Curing of Specimens*—Mold the test bars in accordance with Test Method C 157/C 157M. Mold the cubes in accordance with Test Method C 109/C 109M. A set of specimens to test one cement 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, seal the plate to the mold so as to be watertight, and place the mold in the curing tank in water at 35 ± 3°C (95 ± 5°F) for 23 1/2 h ± 30 min, as in Test Method C 684, Procedure A (Warm-Water Method). Place the sealed molds in the curing tank with the bottom of the bars as cast down, that is, in the same relative position in which the bars were cast. At 23 1/2 h ± 30 min, remove molds from tank 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 C 917) that would justify making fewer cubes, only those needed to confirm the time the mortar achieves 20.0 ± 1.0 MPa (3000 ± 150 psi) are needed.

9.2 *Subsequent Curing and Preparation for Test*—After demolding, store all bars and cubes, except the two to be

⁵ See *Manual of Cement Testing*, Section on Safety, *Annual Book of ASTM Standards*, Vol 04.01.

broken, in a curing tank of saturated limewater at $23.0 \pm 2.0^\circ\text{C}$ ($73.5 \pm 3.5^\circ\text{F}$). Break two cubes in compression in accordance with Test Method C 109/C 109M 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 C 490 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. 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.

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 $23.0 \pm 2.0^\circ\text{C}$ ($73.5 \pm 3.5^\circ\text{C}$). (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 C 511.

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 C 490. Review the data at 15 weeks. Make the subsequent measurements at a minimum of 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 gage 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. When all the bars have been measured, discard the used solution. 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:

ΔL = 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 gage length, or 250 mm (10 in.) as applicable. (See C 490).

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.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 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

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

ANNEX

(Mandatory Information)

A1. Determination of Water Content of Sodium Sulfate

A1.1 Apparatus

A1.1.1 Porcelain crucible, 15mL,

A1.1.2 Oven, drying capable of maintaining $110 \pm 5^\circ\text{C}$

A1.1.3 Desiccator with a good desiccant, such as magnesium perchlorate, or activated alumina. Calcium sulfate treated with a color-change indicator that shows when it has lost its effectiveness is also suitable.

A1.1.4 Balance as described in Section 4 of Test Method C 114.

A1.2 Determining the water content

Determine the mass of approximately 1 g of the sodium sulfate in a tared porcelain crucible to the nearest 0.0001 g. Dry the crucible and its contents for 1 ± 0.25 hour in an oven at a temperature of $110 \pm 5^\circ\text{C}$. Remove the crucible and its contents from the oven and cool in a desiccator until they are

at room temperature. (See Note A1.1) Record the mass of the crucible and sodium sulfate after heating.

NOTE A1.1—15-30 minutes are usually sufficient. A small Pyrex beaker could be used as long as the mass can accurately be weighed (e.g. 50ml beaker).

A1.3 Calculation

Calculate the water content of the sodium sulfate to the nearest 0.1%:

$$\% \text{ WC} = \frac{(M_i - M_f) \times 100}{M_{ss}} \quad (\text{A1.1})$$

where:

% WC = water content, %

M_i = mass of sodium sulfate + crucible before heating,
g

M_f = mass of sodium sulfate + crucible after heating, g
 M_{ss} = mass of sodium sulfate before heating, g

Use the % WC to correct the mass of sodium sulfate used in Section 5.4 (see Note A1.2).

NOTE A1.2—Example: Water content was determined to be 5.0 %. Thus, the corrected mass of anhydrous sodium sulfate present would be:

$$\frac{\text{Mass sodium sulfate uncorrected} \times (100 - \% \text{ WC})}{100} \quad (\text{A1.2})$$

or:

$$\frac{50 \text{ g Na}_2\text{SO}_4 \cdot x \text{ H}_2\text{O} \times (100 - 5)}{100} = 47.5 \text{ g} \quad (\text{A1.3})$$

or in reverse:

$$\frac{50 \text{ g Na}_2\text{SO}_4 \times 100}{(100 - 5)} = 52.6 \text{ g Na}_2\text{SO}_4 \cdot x \text{ H}_2\text{O} \quad (\text{A1.4})$$

APPENDIX

(Nonmandatory Information)

X1. Technical Background

X1.1 Test Method C 1012 was developed to explore the suitability of a test method for sulfate resistance based on length-change measurements of molded prisms immersed in sulfate solution. This approach was taken after it was established that Test Method C 452, in which the sulfate is added to the mortar as it is mixed, and the prisms stored in fresh water, was not able properly to evaluate sulfate resistance of blended cements and blends of portland cement with pozzolan or slag.

X1.2 This test method has been subjected to two rounds of cooperative testing. The first program involved five cements (Types I, II, and V of Specification C 150, and Types IS and IP of Specification C 595). The test solution contained both sodium sulfate and magnesium sulfate, each in the amount of 0.176 mol/L. A report of this work is available. A paper giving results using this test method has been published (1).⁷ The second program involved eight blends using a Type I and a Type II cement with 50 and 70 % slag, 25 % Class F, and 35 % Class C fly ash (Specification C 618). Two test solutions were used: one contained 0.303 mol/L sodium sulfate and 0.049 % magnesium sulfate; the other contained 0.352 mol/L sodium sulfate only.

X1.3 This test method involves length change of hydraulic-cement mortar bars immersed in a sulfate solution. Mortar bars are placed in the sulfate solution after companion mortar cubes have reached a compressive strength of 20.0 ± 1.0 MPa (3000 ± 150 psi). Attempts to use Test Method C 452 for blended cements and blends of portland cement and slag (Specification C 989) or pozzolan (Specification C 618) with portland cement failed to yield results that correlate with field and laboratory experience where the mortar or concrete developed some maturity prior to being exposed to external sulfate solution (2). Since some blended cements and some blends of slag and pozzolan with portland cement have been successfully used where sulfate resistance was needed, they are tested under circumstances which permit comparisons to be based on results obtained when all the sulfate exposures are begun at equivalent

strength since, in practice, it is likely that the concrete will be at about the same strength when sulfate attack begins regardless of the type of cementitious medium employed. This test method is also applicable for use in evaluating portland cements.

X1.4 The solution of 0.176 mol/L of each of the two sulfates (magnesium and sodium) studied in the first cooperative program, is believed likely to adversely affect blends or blended cements containing slag, due to a magnesium ion (3, 4) attack, this attack being unrelated to sulfate attack. Therefore, this solution should not be used to evaluate such systems unless they are to be exposed in service to attack in which the magnesium ion (Mg^{++}) is present in amounts approaching that of the test solution (4200 ppm).

X1.5 This test method does not simulate the mechanism of sulfate attack by solutions of sulfate compositions other than that used. If evaluation of behavior due to exposure to a given sulfate solution is desired, that solution should be used.

X1.6 The user may therefore modify this test method to use any relevant compositions or concentrations of aggressive solutions, other ages or degrees of maturity as the basis for beginning the exposure, mortars of different proportions, or different or additional means of assessing the influence of the aggressive solution on the specimens. Much work was done using mortar bars of lower strength (higher porosity) in previous cooperative tests sponsored by Committee C01 (5-7). A procedure in which sodium sulfate is kept at constant alkalinity/acidity has been proposed (8). Other methods of assessing the influence of the aggressive solutions that have been used or proposed include (a) change in pulse velocity (Test Method C 597), (b) change in resonant frequency (Test Method C 215), (c) change in compressive strength (Test Methods C 109/C 109M and C 349), (d) change in flexural strength (Test Method C 348), (e) change in mass, and (f) change in hardness (Test Methods E 18).

X1.7 Work reported by Polivka and Brown (9) in 1958 and by Mehta and Polivka in 1975 (10) included tests of concrete exposed to a mixed sulfate solution containing 5 % each of

⁷ The boldface numbers in parentheses refer to the list of references at the end of this test method.

sodium and magnesium sulfate. Other relevant work is cited in the references (1-19).

X1.8 The second cooperative study used both a mixed sodium/magnesium sulfate solution and a sodium sulfate

solution. Precision of the results and the ranking of the cements did not differ between the solutions. Hence, the simpler test, using sodium sulfate, was selected as the standard.

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