



Standard Test Method for Approximation of Optimum SO₃ in Hydraulic Cement Using Compressive Strength¹

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1. Scope

1.1 This test method describes the determination of approximate optimum SO₃ for maximum compressive strength at 24 h, 3 days, or 7 days by measuring the change in strength produced in hydraulic cement mortar as a result of substituting calcium sulfate for a portion of the cement.

1.2 This test method refers to the sulfur trioxide (SO₃) content of the cement only. Slag cements and occasionally other hydraulic cements can contain sulfide or other forms of sulfur. The determination of SO₃ content by rapid methods may include these other forms, and may therefore produce a significant error. If a significant error occurs, analyze the cement for SO₃ content using the reference test method of Test Methods C114 for sulfur trioxide.

1.3 Values stated as SI units are to be regarded as standard.

1.4 *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

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

C204 Test Methods for Fineness of Hydraulic Cement by Air-Permeability Apparatus

C305 Practice for Mechanical Mixing of Hydraulic Cement

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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.

Pastes and Mortars of Plastic Consistency

C471M Test Methods for Chemical Analysis of Gypsum and Gypsum Products (Metric)

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

C778 Specification for Sand

C1157 Performance Specification for Hydraulic Cement

C1437 Test Method for Flow of Hydraulic Cement Mortar

3. Significance and Use

3.1 The purpose of this test method is to estimate the SO₃ content for a hydraulic cement that gives maximum compressive strength in mortar made and cured at 23°C. The value obtained is one way to establish an appropriate level of sulfate in the manufacture of cements specified in Specifications C150, C595 and C1157.

3.2 The SO₃ content of a cement giving maximum compressive strength is different at different ages of mortar; typically this SO₃ content is higher at 3 days than the 24-h, and often higher for 7 days than that for 3 days. A manufacturer can choose the age of 24-h, 3 days or 7 days for specimens at which to determine optimum SO₃ content.

3.3 This test method indicates optimum SO₃ content for cement in mortar made and cured at a standard temperature of 23.0 ± 2.0°C (73.5 ± 3.5°F). The optimum SO₃ increases with increasing temperature and may increase when water-reducing admixtures are used.

3.4 It should not be assumed that the optimum SO₃ estimated in this test method is the same SO₃ content for optimum performance of a concrete prepared from the cement.

3.5 The test method is applicable to cements specified in Specifications C150, C595, and C1157.

4. Apparatus

4.1 Use the apparatus as specified in Test Method C109/C109M.

5. Materials

5.1 *Calcium Sulfate*—Use calcium sulfate for addition to the cement that is either a high-grade natural gypsum having an

SO₃ content of at least 46 %, or the calcium sulfate from the source used for the intended plant production. Grind the calcium sulfate to 100 % passing the 75- μ m (No. 200) sieve, and at least 800 m²/kg Blaine fineness (Test Method C204). If the SO₃ content of the calcium sulfate is unknown, analyze it in accordance with Test Methods C471M.

5.2 *Graded Standard Sand*—Use graded sand conforming to Specification C778.

6. Test Specimens

6.1 Make six specimens from each batch of mortar. Make two rounds of three mixtures of mortar as described in 6.3. Make both rounds during the same day if possible. Make only complete rounds on any given day.

6.2 *Preparing Specimen Molds*—Prepare molds in accordance with the section on Preparation of Specimen Molds of Test Method C109/C109M.

6.3 *Proportioning, Consistency, and Mixing of Mortar*—Proportion the dry material for the standard mortar as one part of cement to 2.75 parts graded sand by mass. Mix the following quantities of dry materials at one time for a 6-cube batch:

Mixture No. 1	500 g cement 1375.0 g graded sand
Mixture No. 2	494.6 g cement 5.3 g calcium sulfate 1375.0 g graded sand
Mixture No. 3	489.2 g cement 10.6 g calcium sulfate 1375.0 g graded sand

6.3.1 Use an amount of mixing water to produce a flow of 110 ± 5 using 25 drops of the table as determined in the section on Procedures in Test Method C1437 for all types of cement.

6.3.2 Mix mechanically in accordance with the section on Procedure for Mixing Mortars of Practice C305, except as follows:

6.3.2.1 Add the calcium sulfate to the water; then start the mixer and mix at slow speed (140 ± 5 rpm) for 15 s; then stop the mixer and add the cement to the water; then start the mixer and mix at slow speed (140 ± 5 rpm) for 30 s.

6.4 *Molding of Test Specimens*—Mold specimens in accordance with the Procedure Section of Test Method C109/C109M.

6.5 *Storage of Test Specimens*—Immediately upon completion of molding, place the test specimens in the moist closet or moist room. Keep all test specimens, immediately after molding, in the molds on the base plates in the moist closet or moist room for 20 to 24 h with their upper surfaces exposed to the moist air but protected from dripping water. If the specimens are removed from the molds before 24 h, keep them on the shelves of the moist closet or moist room until they are 24-h old and then immerse the specimens, except those for the 24-h test, in saturated lime water in storage tanks constructed of noncorroding materials until the age chosen for the test. Keep the storage water clean by changing as required.

7. Temperature and Humidity

7.1 The temperature and humidity of the moist cabinet or moist room shall be as specified in Specification C511.

7.2 The temperature and humidity of the mixing room shall be as specified in Test Method C109/C109M.

8. Procedure

8.1 Test the specimens at the age of $24 \pm \frac{1}{4}$ h, 3 days ± 1 h, or 7 days ± 3 h, from the time the cement and water made contact during mixing, immediately after removal from storage in accordance with Test Method C109/C109M. If more than one specimen at a time is removed from storage, keep these specimens covered with a damp cloth until time of testing. Note and observe precautions regarding testing and retesting given in the applicable sections of Test Method C109/C109M.

9. Calculation

9.1 Calculate and record the strength of specimens as described in the section on Calculation in Test Method C109/C109M.

9.1.1 Calculate the indicated optimum SO₃ content as follows:

$$G = [a/(a - b)]c + d + c/2$$

where:

- G = optimum percent SO₃,
- a = average strength both rounds of Mixture No. 2 minus Mixture No. 1,
- b = average strength both rounds of Mixture No. 3 minus Mixture No. 2,
- c = (percent SO₃ in calcium sulfate)/100, and
- d = percent SO₃ in test cement.

10. Retest

10.1 Regard the results obtained as suspect and repeat the test under any of the following conditions:

- 10.1.1 If a and b both are positive and the ratio a/b is less than 2.00,
- 10.1.2 If a and b both are negative and the ratio a/b is greater than 0.500, or
- 10.1.3 If a is negative and b is positive.

11. Report

11.1 If repeat tests confirm that the ratio of a/b departs from the limiting values as stated above, report the SO₃ content of the cement as “above optimum” or “below optimum” as the data may indicate, but without stating the numerical value of optimum SO₃.

11.2 If the test data indicate that the cement is “below optimum” but do not permit a computation of the percentage of SO₃ required for optimum, an additional series of tests involving greater substitutions of gypsum for part of the cement should yield results that will permit an acceptable calculation of the percentage of SO₃ required for optimum. If the test data indicate that the cement is “above optimum” but do not permit a computation of the percentage of SO₃ required for optimum, in order to establish the required percentage of SO₃ for

optimum, it is necessary to make tests of a new sample of similar cement but having an original SO_3 content lower than that of the original cement.

11.3 Report the age of the test specimens.

12. Precision and Bias

12.1 *Precision*—The coefficient of variation of strength values for this test method, using 6 cubes per batch, is very similar to that for Test Method **C109/C109M** compression cubes tested at later ages. Refer to Test Method **C109/C109M**

for suitable values. No data currently exist for the precision of the estimate for optimum sulfate content.

12.2 *Bias*—Since there is no accepted reference material suitable for determining optimum SO_3 , bias has not been determined.

13. Keywords

13.1 blended hydraulic cement; calcium sulfate; cement; compressive strength; gypsum; hydraulic cement; optimum sulfate content (of cement); portland cement; strength (of cement); sulfate content (of cement)

APPENDIX

(Nonmandatory Information)

X1. DISCUSSION OF THE TERM “OPTIMUM”

X1.1 The scope statement notes that this test method determines the optimum SO_3 content in cements in mortar at a particular temperature and age. Usually, but not always, the SO_3 content that produces the highest 24-h strength at 23°C also produces approximately the lowest expansion in water and the lowest contraction in air at that temperature.

X1.2 The “optimum” determined by this test is approximate. The “optimum” SO_3 content will vary with changes in mortar proportions; between cement paste, mortars and concrete; will vary with the source of SO_3 ; with the age of test; with the use of chemical admixtures; and with the use of supplementary cementitious materials. Thus, the term “optimum SO_3 ” refers to an approximate value.

X1.3 The age for determining the optimum is typically chosen by the manufacturer based on experience with local concrete materials.

X1.4 For convenience, the method uses the compressive strength of specimens at three different SO_3 contents for the cement. If the strength results are plotted against SO_3 content, the three points are assumed to give a curve in the shape of a parabola, and the calculation used assumes this shape. If more points are used with smaller SO_3 content increments, the shape of the curve is seen to be that of a “sawtooth,” with a decreasing slope up to the apex at maximum strength, followed by a precipitous fall in strength immediately after the apex. The apex represents the actual optimum SO_3 content, and is typically higher than that calculated using three points. Therefore, this affords a small “cushion” when targeting the calculated optimum SO_3 (determined using three points) during manufacture to allow for variation in the process.

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