Designation: C 265 – 03a

Standard Test Method for Water-Extractable Sulfate in Hydrated Hydraulic Cement Mortar[1]

This standard is issued under the fixed designation C 265; 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 measurement of water-extractable SO_3 in hardened hydraulic cement mortar. This measurement is assumed to represent unreacted, available sulfate remaining in the mortar.

1.1.1 (**Warning:**—Fresh hydraulic cementious mixtures are caustic and may cause chemical burns to skin and tissue upon prolonged exposure.)

1.2 The values stated in SI units are to be regarded as the standard. The values given in parentheses are for information only.

1.3 Values in SI units were obtained by measurement in SI units or by appropriate conversion using the Rules for Conversion and Rounding given in IEEE/ASTM SI-10 Standard of measurements made in other units.

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:*

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[1] This test method is under the jurisdiction of ASTM Committee C01 on Cement and is the direct responsibility of Subcommittee C01.28 on Sulfate Content.
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C 109/C 109M Test Method for Compressive Strength of Hydraulic Cement Mortars (Using 2-in. or [50-mm] Cube Specimens)[2]
C 114 Test Methods for Chemical Analysis of Hydraulic Cement[2]
C 150 Specification for Portland Cement[2]
C 219 Terminology Relating to Hydraulic Cement[2]
C 305 Practice for Mechanical Mixing of Hydraulic Cement Pastes and Mortars of Plastic Consistency[2]
C 595 Specification for Blended Hydraulic Cements [2]
C 778 Specification for Standard Sand[2]
C 1157 Performance Specification for Hydraulic Cement [2]
D 1193 Specification for Reagent Water[3]
E 11 Specification for Wire-Cloth and Sieves for Testing Purposes[4]
IEEE/ASTM SI-10 Standard for Use of the International System of Units (SI): The Modern Metric System[4]

3. Terminology

3.1 *Definitions*— The terms used in this test method are defined in accordance with Terminology C 219.

4. Significance and Use

4.1 Excess soluble sulfate ions in hardened cement can result in reduced durability. This test method is intended ~~for use by manufacturers of hydraulic cement and those interested in research on a suitable method for determining~~ to show whether calcium sulfate has ~~or has not~~ been ~~added~~ used in ~~an~~ cement in such amount ~~considered~~ to ~~be optimum. Also,~~ leave excess soluble sulfate in hardened mortar. The test method is used to establish compliance in Specification C 595 for ~~any such cement having an above optimum~~ those cements in which optimized SO_3 ~~content, this test method establishes whether that excess~~ exceeds the ~~limit allowed in Specification C 595.~~ table limit. This test method also can provide useful information on other hydraulic cements, such as those specified in Specifications C 150 and C 1157.

5. Apparatus

5.1 *Sieve*—A 2.36-mm (No.8) sieve conforming to Specification E 11.
5.2 *Mixer, Bowl, and Paddle*—An electrically driven mechanical mixer equipped with a paddle and bowl, as specified in the Apparatus section of Practice C 305.
5.3 *Polyethylene Containers*—Watertight polyethylene bags of 1-L (1-qt) capacity or approximately 360-mm (14-in.) sheet material, made using polyethylene at least 0.10-mm (0.004-in.) in thickness.
5.4 *Mortar and Pestle*—A mortar of 1.5-L (1½-qt) size, and a pestle, both of which shall be iron or porcelain.
5.5 *Water Bath*—A water bath thermostatically controlled at 23.0 ± 0.15°C .

6. Reagents and Materials

6.1 *Mixing Water*— Reagent water conforming to the numerical limits of Type II of Specification D 1193.
6.2 *Graded Sand*— Sand conforming to the requirements for graded sand in Specification C 778.

7. Temperature and Relative Humidity

7.1 Maintain the temperature of the molding room at 23.0 ± 2.0°C . Adjust the temperature of the dry materials and the mixing water, prior to mixing, so that the temperature of the mortar, immediately upon completion of mixing is 23 ± 1°C .
7.2 Maintain the relative humidity of the laboratory so that it is not less than 50 %.

8. Preparation of Test Mortar

8.1 Proportion the mortar in accordance with the instructions for a six-cube batch given in the section on Composition of Mortars of Test Method C 109/C 109M, except use a water-cementious material ratio of 0.5, by mass. Mix mechanically in accordance with Procedure for Mixing Mortars of Practice C 305.
8.2 Split the batch immediately after mixing then place approximately 450 g of mortar in each of the two polyethylene containers. Obtain the temperature of each portion to ensure that it is within the range 23 ± 1°C . If bags are used, twist the neck of each and seal with a rubber band; double the neck over and seal with a second band. If sheets are used to contain the samples, gather the corners and edges to make a bag and seal in similar manner.

9. Storage of Test Mortar

9.1 Immediately immerse both containers in the water bath maintained at (23.0 ± 0.15°C) .

[2] *Annual Book of ASTM Standards*, Vol 04.01.
[3] *Annual Book of ASTM Standards*, Vol 11.01.
[4] *Annual Book of ASTM Standards*, Vol 14.04.

10. Preparation of Cement Extract

10.1 At 24 ± ¼ h after the instant the cement and water were mixed together, remove the containers successively from the water bath, pulverizing each sample in turn with the pestle in the mortar as rapidly as possible. Immediately upon removal from the water bath treat each sample individually as follows: First pulverize approximately one third of the specimen until most of it passes the 2.36-mm (No. 8) sieve. Return the coarse residue to the mortar and grind another one third of the specimen as before. Repeat the process until the entire specimen is pulverized to pass the 2.36-mm (No. 8) sieve. Place 400 g of the pulverized material in a 600-mL beaker and add 100 mL of water at a temperature of 23.0 ± 0.6°C . Quickly mix to uniform consistency with a spatula; then stir mechanically for 2 min. Use a stirrer of such a type and speed that all particles of the slurry are suspended, with no settling and no appreciable increase of the temperature of the slurry occurs.

10.2 Filter the slurry on a dry No. 2A Büchner funnel with the aid of suction using a dry 90-mm, medium, ashless paper. Complete filtration within a period of 2 min. Filter again, whether the filtrate is turbid or not, using a new dry filter paper without suction.

11. Analysis of the Extract

11.1 Transfer 50.0 mL of the clear extract by means of a 50-mL pipet to a 400-mL beaker, dilute with distilled water to about 250 mL, and add 5 mL of concentrated hydrochloric acid (HCl, sp gr 1.19). Heat to boiling, and proceed in accordance with the sulfur trioxide determination section of Test Methods C 114. Perform a blank on the reagents in use in the analysis.

11.1.1 After the $BaCl_2$ solution is added in accordance with the sulfur trioxide determination section of Test Methods C 114, boil the solution vigorously for 15 min.

11.2 When the amount of filtrate is insufficient to provide a 50-mL aliquot, use 25 mL of the extract and a factor twice that given in Section 12.

12. Calculation and Report

12.1 Calculate the SO_3 content to the nearest 0.01 g/L of solution, as follows:

$$SO_3, g/L = W \times 6.86 \quad (1)$$

where:

W = grams of barium sulfate ($BaSO_4$) precipitated.

12.2 Report the average of the two determinations.

13. Precision and Bias

13.1 *Precision*—The single-operator (within-laboratory) standard deviation has been found to be 0.077 g/L SO_3 throughout the range of 0.20 to 0.85 g/L SO_3. Therefore, results of two properly conducted tests by the same operator on water-extractable SO_3 in hardened hydraulic cement mortars of the same material should not differ from each other by more than 0.22 g/L SO_3. The multi-laboratory (between-laboratory) standard deviation has been found to be 0.137g/L SO_3 throughout the range of 0.20 to 0.85 g/L SO_3. Therefore, results of two properly conducted tests on the same material, from two different laboratories on water-extractable SO_3 in hardened hydraulic cement mortars should not differ from each other by more than 0.39 g/L SO_3 .

13.2 *Bias*—No justifiable statement can be made on the bias of the procedure in this test method for measuring the water-extractable SO_3 in hardened hydraulic cement mortars, because no reference samples are available.

14. Keywords

14.1 hydraulic cement; optimum sulfate content; sulfate; sulfate content

For additional useful information on details of cement test methods, reference may be made to the "Manual of Cement Testing," *Annual Book of ASTM Standards,* **Vol 04.01.**