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

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1. Scope-Scope*

- 1.1 This guide describes the determination of approximate optimum SO₃ for maximum eompressive strength at 24 h, 3 days, or 7 days by measuring the change in strength produced in hydraulic cement mortar performance as a result of substituting calcium sulfate for a portion of the cement.
- 1.2 This guide refers to the sulfur trioxide (SO_3) content of the cement only. Slag cements and occasionally other hydraulic cements can contain sulfide or other forms of sulfur. The determination of SO_3 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 SO_3 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

C39/C39M Test Method for Compressive Strength of Cylindrical Concrete Specimens

C78 Test Method for Flexural Strength of Concrete (Using Simple Beam with Third-Point Loading)

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

C192 Practice for Making and Curing Concrete Test Specimens in the Laboratory

C204 Test Methods for Fineness of Hydraulic Cement by Air-Permeability Apparatus 92795bd2c403/astm-c563-17

C305 Practice for Mechanical Mixing of Hydraulic Cement Pastes and Mortars of Plastic Consistency

C430 Test Method for Fineness of Hydraulic Cement by the 45-µm (No. 325) Sieve

C465 Specification for Processing Additions for Use in the Manufacture of Hydraulic Cements

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

C511C595 Specification for Mixing Rooms, Moist Cabinets, Moist Rooms, and Water Storage Tanks Used in the Testing of Hydraulic Cements and Concretes Blended Hydraulic Cements

C595C596 Specification for Blended Hydraulic Cements Test Method for Drying Shrinkage of Mortar Containing Hydraulic Cement

C1157 Performance Specification for Hydraulic Cement

C1437 Test Method for Flow of Hydraulic Cement Mortar

C1702 Test Method for Measurement of Heat of Hydration of Hydraulic Cementitious Materials Using Isothermal Conduction Calorimetry

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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. Significance and Use

- 3.1 The purpose of this guide is to estimate the SO₃ content for a hydraulic cement that gives maximum empressive strength in mortar made and cured at 23°C. performance. 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 performance is different at different ages of mortar; typically this SO_{ages}, with different performance criteria 3 content is higher at 3 days than the 24-h, and often higher for 7 days than that for 3 days.and with different materials such supplementary cementitious materials and chemical admixtures. A manufacturer can choose the age of 24-h, 3 daysperformance criteriato determine optimum SO₃ or 7 days for content. This optimum SO₃ specimens at which to determine optimum SO_{content} may be a compromise between different ages and different performance criteria.

Note 1—Typically, the optimum SO₃ content is higher the later the age.

- 3.3 This guide indicates optimum SO_3 content for cement in mortar made and cured at a standard temperature of 23.0 ± 2.0 °C (73.5 \pm 3.5°F). The optimum SO_3 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 guide is the same SO₃ content for optimum performance of a concrete prepared from the cement.
 - 3.5 The guide is applicable to cements specified in Specifications C150, C595, and C1157.

4. Apparatus

4.1 Use the apparatus as specified in Test Method Methods C109/C109M, C192, C596, or C1702.

5. Materials

5.1 Calcium Sulfate—Use calcium sulfate for addition to the cement that is either a high-grade natural gypsum having an SO_3 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 SO_3 content of the calcium sulfate is unknown, analyze it in accordance with Test Methods C471M.

Note 2—The calcium sulfate source can impact the optimum sulfate result due in part to differences in form of the calcium sulfate (for example, gypsum, calcium sulfate hemi-hydrate, or anhydrous calcium sulfate). Temperatures in cement finish mills during production can reach levels to partially or completely change the form of calcium sulfate in cement.

- 5.2 Cement—Make cements of different sulfate levels at a single production site. Make the cements so that the amount of calcium sulfate added, and the subsequent dilution effects, are the only difference in constituent materials.
- 5.2.1 Grind samples to a fineness within 13 m/kg of the other samples when tested in accordance with Test Method C204. Since calcium sulfate sources are typically softer than clinker, an adjustment of 10 m²/kg for every 1 % calcium sulfate addition is permitted, as shown in equation Eq 1.

$$F_{AX} = F_{MX} - \frac{10 \cdot (SO_{3X} - SO_{3,median})}{SO_{3CS}/100}$$
 (1)

where:

 $SO_{3,CS}$ = percentage of SO_3 in the calcium sulfate,

 $SO_{3,median} = SO_3$ percentage of the sample with the median SO_3 of the samples tested,

 $SO_{3,X}$ = SO_3 percentage of cement sample X, $F_{M,X}$ = measured fineness of cement sample X, and adjusted fineness of cement sample X.

Note 3—Differences in the mill conditions between samples of different sulfate levels should be minimized. For this reason samples are normally taken during the same production campaign. Strategies should be employed to minimize the differences in fineness of the clinker when taking samples, such as targeting a specific sieve size range and adjusting around that target within reasonable tolerances. Since calcium sulfate is softer, and thus easier to grind than clinker, increases in calcium sulfate content will elevate the fineness of the cement without a change in the grinding energy or the fineness of the clinker.

Note 4—As an example, consider the case of one cement sample with an SO_3 content of 2.7 % and a fineness of 380 m²/kg, which is the sample with the median SO_3 content, and another sample with an SO_3 content of 3.7 % and a fineness of 405 m²/kg. The second sample has a 1.0% higher SO_3 content, or 2.2 % more calcium sulfate addition, assuming the calcium sulfate was 45 % SO_3 . The adjusted cement fineness of the second sample would be reduced by 22 m²/kg (10 × 2.2) to 383 m²/kg by using Equation Eq 1 as shown in Eq 2. This value of 383 m²/kg is within 13 m²/kg of the fineness of 380 m²/kg, and thus is acceptable for testing.

$$F_{AX} = 405 - \frac{10 \cdot (3.7 - 2.7)}{\frac{45}{100}} = 383 \, m^2 / kg \tag{2}$$

5.2.2 Determine the percentage of the following analytes by Test Method C114 for each cement tested: silicon dioxide (SiO₂), aluminum oxide (Al₂O₃), ferric oxide (Fe₂O₃), calcium oxide (CaO), magnesium oxide (MgO), sulfur trioxide (SO₃), loss on

ignition, insoluble residue, sodium oxide (Na_2O), and potassium oxide (K_2O). Calculate the potential percentages of the following compounds for portland cements according to Specification C150: tricalcium silicate, dicalcium silicate, tricalcium aluminate and tetracalcium aluminoferrite. When applicable, report the amount of limestone and Specification C465 inorganic processing additions according to Specification C150. Determine the fineness of each cement tested according to Test Methods C204 and C430.

Note 5—The amount of material retained on the 45-μm sieve has been used as an indication of the clinker fineness. When high efficiency separators are used, the amount retained on a 20-μm sieve has also been used as an indicator of clinker fineness.

6. Procedure

- 6.1 Sulfate Levels to Test—Test at least five different sulfate levels.
- 6.1.1 SO₃ contents are to be at least 0.20 % different unless more than five different SO₃ contents are being tested. The maximum and minimum SO₃ content of the blended samples must differ by at least 2.0 % SO₃ content.

Note 6—The same mixture design and materials shall be used when comparing different SO₃ contents. Use one or more of the following test methods to evaluate the performance:

- 6.1.1.1 When adding calcium sulfate it is considered as part of the mass of cement for proportioning.
- 6.1.1.2 Use the following equation to calculate the total SO₃ in the blended sample of cement and calcium sulfate:

$$SO_{3-Total} = \frac{M_{calcium sulfate}}{M_{calcium sulfate} + M_{comennt}} \times SO_{3-calcium sulfate} + \frac{M_{cement}}{M_{calcium sulfate} + M_{comennt}} \times SO_{3-cement}$$
(3)

where:

 $\underline{M_{calcium\ sulfate}}$ = the mass of the calcium sulfate,

 $\underline{M_{cement}}$ = the mass of the cement,

 $SO_{3-cement sulfate}$ = the percent by mass of SO_3 in the calcium sulfate, and

 $SO_{3-cement}$ = the percent by mass of the SO_3 in the cement.

Note 7—More sulfate levels may be tested to help improve the precision of the interpretation of the results. Extremely high and low sulfate levels can give results that deviate from the typical peak behavior which may need to be treated as outliers when using a mathematical fitting procedure.

- 6.2 Determine mortar compressive strength at each sulfate level at the age of 24 The same mixture design and materials shall be used when comparing different SO_3 \pm contents. $\frac{1}{4}$ h, 3 days \pm 1 h, or 7 days \pm 3 h in accordance with Test Method Use one or more of the following test methods to evaluate the performance: C109/C109M except as follows:
- 6.2.1 Mortar compressive strength—Determine mortar compressive strength at each sulfate level at the age of $24 \pm \frac{1}{4}$ h, 3 days \pm 1 h, or 7 days \pm 3 h in accordance with Test Method C109/C109M except as follows:
- 6.2.1.1 When mixing in accordance with the "Procedure for Mixing Mortars" section of Practice C305, add the calcium sulfate to the water, unless the calcium sulfate addition has been previously ground and mixed with the cement; 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.
- 6.2.1.2 Use the amount of mixing water to produce a flow of 110 ± 5 for one of the mixtures using 25 drops of the table as determined in the section on Procedures in Test Method C1437. Use that same amount of water (constant w/cm) for each mixture with different sulfate levels.
 - Note 8—The mixture with the median sulfate level or lowest sulfate level is often used to determine the water content.
- 6.2.2 Heat of hydration—Determine heat of hydration at each sulfate level at the age of $24 \pm \frac{1}{4}$ h, 3 days ± 1 h, or 7 days ± 3 h in accordance with Test Method C1702 except as follows:
- 6.2.2.1 Add the calcium sulfate to the water, unless the calcium sulfate addition has been previously ground and mixed with the cement;
- 6.2.2.2 Additions of other materials typically used in concrete, such as supplementary cementitious materials and chemical admixtures, can be used.
- 6.2.2.3 Mortars are allowed to be used in addition to pastes. When testing with mortars use the same sand content for each different mixture.
 - 6.2.2.4 Testing at temperatures besides 23°C is allowed. Use the same temperature for each different mixture.
 - 6.2.3 Concrete Strength—Prepare all material according to Practice C192 except as follows:
- 6.2.3.1 Add the calcium sulfate to the water, unless the calcium sulfate addition has been previously ground and mixed with the cement.
- <u>6.2.3.2</u> When applicable, determine compressive strength according to Test Method C39/C39M. When applicable, determine flexural strength according to Test Method C78.
- 6.2.3.3 Testing at concrete and curing temperatures other than specified is allowed. Use the same material temperature (all mixtures within 10°C range) and the same curing temperature (all curing temperatures within 4°C range) for each of the different mixtures