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Standard Guide for Approximation of Optimum SO_3 in Hydraulic Cement[1]

This standard is issued under the fixed designation C563; 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 guide describes the determination of approximate optimum SO_3 for maximum 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
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)
C595 Specification for Blended Hydraulic Cements
C596 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

3. Significance and Use

3.1 The purpose of this guide is to estimate the SO_3 content for a hydraulic cement that gives maximum 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 SO_3 content of a cement giving maximum performance is different at different ages, with different performance criteria and with different materials such supplementary cementitious materials and chemical admixtures. A manufacturer can choose the performance criteriato determine optimum SO_3 content. This optimum SO_3 content may be a compromise between different ages and different performance criteria.

NOTE 1—Typically, the optimum SO_3 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 ± 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 SO_3 estimated in this guide is the same SO_3 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.

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[1] This guide 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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[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.

***A Summary of Changes section appears at the end of this standard**

4. Apparatus

4.1 Use the apparatus as specified in Test 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-µm (No. 200) sieve, and at least 800 m^2/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 m^2/kg for every 1 % calcium sulfate addition is permitted, as shown in equation Eq 1.

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

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
$F_{A,X}$ = 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 m^2/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 m^2/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 m^2/kg (10 × 2.2) to 383 m^2/kg by using Equation Eq 1 as shown in Eq 2. This value of 383 m^2/kg is within 13 m^2/kg of the fineness of 380 m^2/kg, and thus is acceptable for testing.

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

5.2.2 Determine the percentage of the following analytes by Test Method C114 for each cement tested: silicon dioxide (SiO_2), aluminum oxide (Al_2O_3), ferric oxide (Fe_2O_3), calcium oxide (CaO), magnesium oxide (MgO), sulfur trioxide (SO_3), 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 SO_3 contents are to be at least 0.20 % different unless more than five different SO_3 contents are being tested. The maximum and minimum SO_3 content of the blended samples must differ by at least 2.0 % SO_3 content.

NOTE 6—The same mixture design and materials shall be used when comparing different SO_3 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 SO_3 in the blended sample of cement and calcium sulfate:

$$SO_{3\text{-}Total} = \frac{M_{calcium\ sulfate}}{M_{calciumsulfate} + M_{cememnt}} \times SO_{3\text{-}calcium\ sulfate} + \frac{M_{cement}}{M_{calciumsulfate} + M_{cement}} \times SO_{3\text{-}cement} \qquad (3)$$

where:

$M_{calcium\ sulfate}$ = the mass of the calcium sulfate,
M_{cement} = the mass of the cement,
$SO_{3\text{-cement sulfate}}$ = the percent by mass of SO_3 in the calcium sulfate, and
$SO_{3\text{-cement}}$ = 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 The same mixture design and materials shall be used when comparing different SO_3 contents. Use one or more of the following test methods to evaluate the performance:

6.2.1 *Mortar compressive strength*—Determine mortar compressive strength at each sulfate level at the age of 24 ± ¼ h, 3 days ± 1 h, or 7 days ± 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 ± ¼ 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.

6.2.3.2 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

6.2.4 *Drying Shrinkage of Mortar*—Prepare all material according to Practice C596 except as follows:

6.2.4.1 When mixing in accordance with the section on Procedure for Mixing Mortars 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.

6.2.4.2 Instead of using the amount of mixing water sufficient to produce a flow of 110 ± 5, 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 9—The mixture with the median sulfate level or lowest sulfate level is often used to determine the water content.

7. Interpretation of Results

7.1 Approximate the SO_3 content which gives the maximum performance by one of the following methods:

NOTE 10—See the appendix for an example of how this interpretation is done for each method described below. Depending on which method is chosen the results may differ.

7.1.1 *Visual Fit*—Plot the performance level versus SO_3 content and interpolate the sulfate level at the peak.

7.1.2 *Least Squares Parabolic Fit.*

7.1.2.1 Determine the equation of a least squares fit according to follow equation:

$$\text{Performance Level} = a(\text{SO}_3)^2 + b\text{SO}_3 + c$$

where *a*, *b*, and *c* are fitting coefficients.

NOTE 11—Spreadsheet and graphing programs have the capability to calculate the least squares parabolic fit.

7.1.2.2 Approximate the optimum SO_3 by calculating vertex of the parabolic least squares fit from the following equation:

$$\text{Optimum SO}_3 \text{ approximation} = -b/(2a)$$

where *a* and *b* are coefficients of the parabolic least squares fit.

7.1.3 *Asymmetric Fit*—In cases where the performance level versus SO_3 is skewed to the right or left of the peak a fit using an asymmetric distribution function may provide a better fit than parabolic fit.

NOTE 12—Mathematical and statistics software programs are useful in doing such fits.

NOTE 13—The sulfate level for the maximum performance may or may be not the SO_3 content that one of the tests was conducted at.

8. Retest

8.1 If the approximate optimum sulfate level is greater or lower than all the SO_3 contents tested, then test at additional sulfate levels until at least one SO_3 content is greater than or less than approximate optimum SO_3 content. Repeat the interpretation of results in Section 7 and report on that final set of results.

9. Report

9.1 Report the method(s) and ages used to determine performance

9.2 Report any variations in the method(s) from the standard

9.3 Report of the approximate optimum SO_3 value as required.

9.4 Report if calcium sulfate was added to the cement samples to achieve various levels of SO_3.

9.5 Report the results of chemical and physical analysis, as required by 5.2.2, for the cement sample(s) used.

10. Keywords

10.1 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)