Designation: ~~C563 – 19~~ C563 – 20

Standard Guide for
Approximation of Optimum SO$_3$ in Hydraulic Cement[1]

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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.*

1.5 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

C39/C39M Test Method for Compressive Strength of Cylindrical Concrete Specimens
C78/C78M 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/C150M Specification for Portland Cement
C192/C192M 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/C595M Specification for Blended Hydraulic Cements
C596 Test Method for Drying Shrinkage of Mortar Containing Hydraulic Cement
C1157/C1157M Performance Specification for Hydraulic Cement
C1437 Test Method for Flow of Hydraulic Cement Mortar
C1679 Practice for Measuring Hydration Kinetics of Hydraulic Cementitious Mixtures Using Isothermal Calorimetry
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/C150M, C595/C595M, and C1157/C1157M.

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

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, optionally in presence of supplementary cementitious materials and admixtures. Several alternate methods are allowed: compressive strength of concrete or mortar, heat of hydration of paste or mortar, and drying shrinkage of mortar.

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 in the field using the same materials. The optimum SO_3 is influenced by parameters such as ambient and fresh concrete temperature, admixtures, and supplementary cementitious materials.

3.5 The guide is applicable to cements specified in Specifications C150/C150M, C595/C595M, and C1157/C1157M.

4. Apparatus

4.1 Use the apparatus as specified in Test Methods C109/C109M, C192/C192M, 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~~ 75 µm (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 surface area and 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, while laboratory grinding of calcium sulfate may alter its reactivity through increased fineness and partial change of the form of the calcium sulfate. Different clinkers may react differently to the effect of sulfate forms. In particular, if the dissolution rate of sulfate during cement hydration is at any time lower than the sulfate consumption rate of the aluminates, the sulfate in solution may become temporarily depleted, resulting in elevated aluminate reactivity and significant reductions in alite reactivity. Elevated aluminate reactivity may lead to loss of workability and admixture performance as well as reduced rate of strength development and setting behavior that has a relatively low degree of repeatability and reproducibility. On the other hand, if the dissolution rate of sulfate before placing of concrete is higher than the sulfate consumption rate of the aluminates, precipitation of secondary gypsum may cause loss of workability and false set.

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 m²/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 Eq 1.

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

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where:

~~$SO_{3,CS}$~~	=	~~percentage of SO_3 in the calcium sulfate,~~
$SO_{3,CS}$	=	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,~~
$SO_{3,median}$	=	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 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%~~ 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 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.

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

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

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/C150M: 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/C150M. 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~~ 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~~ 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 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:

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

$$SO_{3\text{-}total} = \frac{M_{calcium\,sulfate}}{M_{calcium\,sulfate} + M_{cement}} \times SO_{3\text{-}calcium\,sulfate} + \frac{M_{cement}}{M_{calcium\,sulfate} + M_{cement}} \times SO_{3\text{-}cement} \qquad (3)$$

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—As an example, consider the case of a cement with a SO_3 content of 2.7 % and calcium sulfate with SO_3 content of 45 % where 594 g of cement are mixed with 6 g calcium sulfate. The total SO_3 of the blend of cement and calcium sulfate would be 3.1 % as shown in equation Eq 4 below:

$$SO_{3\text{-}Total} = \frac{6}{6+594} \times 45\% + \frac{594}{6+594} \times 2.7\% = 3.1\% \qquad (4)$$

$$SO_{3\text{-}total} = \frac{6}{6+594} \times 45\% + \frac{594}{6+594} \times 2.7\% = 3.1\% \qquad (4)$$

Note 8—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—~~ *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 105 to 115 % 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.

6.2.2 *Heat of hydration—Hydration—*Determine heat of hydration at each sulfate level at the desired ages in accordance with Test Method C1702 except as follows:

6.2.2.1 If making calcium sulfate powder additions to an industrially produced cement sample, add the cement and calcium sulfate powder directly into the calorimetry sample vial and dry mix the solids inside the calorimetry sample vial to ensure that the calcium sulfate is blended with the cement prior to water addition. If using industrially-produced cement samples with the calcium sulfate additions already interground, follow the procedure for Test Method C1702 Method A or B except as follows. Alternatively, produce a mortar as described 6.2.1.

6.2.2.2 Additions of other materials typically used in concrete, such as supplementary cementitious materials and chemical admixtures, can be used, provided that the content of supplementary cementitious materials, chemical admixtures, or both are kept the same for each different mixture.

6.2.2.3 When testing with mortars use the same sand content for each different mixture.

6.2.2.4 Testing at temperatures besides ~~23°C~~23 °C is allowed. Use the same temperature for each different mixture.

Note 10—Experience shows that mixing cement paste externally and transferring a subset of paste or mortar to the sample vial generates higher variability because of increased variability of the masses of cement and calcium sulfate transferred from the external mixing vial to the sample vial. If using cement paste with additions of calcium sulfate powder not already interground in the cement, it is recommended to weigh and mix all materials inside the sample vial without removing any material from the sample vial after mixing.

Note 11—Blending of two industrially produced cement samples as described in 5.2, produced within the same day with a low and a high SO_3 content to obtain a series of sub samples with varying SO_3 contents, is known to give good results, provided that the clinker fineness and the clinker reactivity are similar. Alternatively, the addition of calcium sulfate powder to a single industrial cement sample allows for testing the effect of different sulfate forms and may yield a less variable approximate optimum sulfate level as the sulfate level is the only parameter that varies in the test.

6.2.2.5 Any thermal power measured prior to the minimum at point (B) observed prior to the onset of the main hydration peak (C), Fig. 1, shall be excluded from the calculated heat of hydration.

Note 12—Experience shows that optimum SO_3 estimated at a given age using isothermal conduction calorimetry with cement paste is similar to the optimum SO_3 estimated using compressive strength of mortar in ~~Standard~~ Guide C1679. The early heat measurements before the minimum B, Fig. 1, are typically subject to high levels of variability and influenced by relatively small differences in calorimeter and material temperatures, and are therefore excluded.

6.2.2.6 *Composition of Test Mixture—*Adjust the mass of solids and water as needed to obtain a suitable mix size, with the optional addition of sand and other materials typically used in concrete. Ensure that the total masses of cement and cement plus calcium sulfate differ by no more than 5 % within a given test series. Weigh or determine volumetrically a fixed and sufficient amount of water to completely wet and easily mix each individual mixture.

6.2.2.7 Use the same mixing method for all samples to be compared.

Note 13—Several paste mixing methods are known to give suitable results, including: Internal mixing using a disposable impeller left inside the sample vial after mixing; external mixing using a disposable spoon left inside the sample vial after mixing; external mixing using vibration or ultrasound contacting the outside of the sample vial, external mechanical mixing of paste or mortar which is then added to the sample vial. However, better
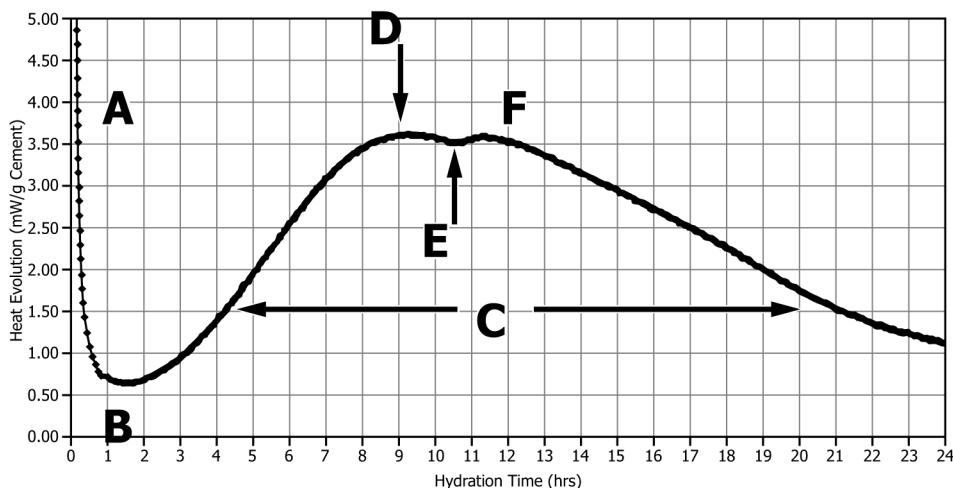
(A) Initial thermal power by dissolution of cement and initial cement hydration; (B) Dormant period associated with very low thermal power indicating slow and well-controlled hydration; (C) Main hydration peak associated mainly with hydration reactions contributing to setting and early strength development, with maximum at (D); and (E) Sulfate depletion point, followed by (F) Accelerated calcium aluminate activity. In this example the approximate time of onset of the main hydration peak is 1 h 45 min.

FIG. 1 ~~(A) initial thermal power by dissolution of cement and initial cement hydration; (B) dormant period associated with very low thermal power indicating slow and well-controlled hydration; (C) main hydration peak associated mainly with hydration reactions contributing to setting and early strength development, with maximum at (D); and (E) sulfate depletion point, followed by (F) accelerated calcium aluminate activity. In this example the approximate time of onset of the main hydration peak is 1 h 45 min.~~Heat Evolution Over Hydration Time

reproducibility can be achieved when the mixing is done within the calorimeter sample vial, as error due to different sampling from a separate mixing bowl to sample vial is avoided, and mixing temperature conditions are easier to control within the sample vial.

6.2.2.8 *Test Duration*—Continue collecting calorimetry data for the period over which sulfate optimization is desired to be achieved.

NOTE 14—Generally optimum SO_3 levels are found to be higher when the degree of cement hydration is higher. Accordingly it is useful to perform this test for several days so that the change in optimum with a cement from a particular clinker source and sulfate source and form can be better understood.

6.2.2.9 Calculate the heat of hydration normalized per gram of cement (any calcium sulfate addition is considered as part of the cement) as described in the section on Calculation in Test Method C1702 with the following exceptions:

(1) Plot the thermal power per gram of cement as a function of time. Determine the time at which the minimum B, Fig. 1, occurs for each ~~sample~~sample.

(2) Remove all such data points before the minimum B, Fig. 1, at the time of onset of the main hydration peak before calculating the heat of hydration normalized per gram cement.

NOTE 15—It is important to use the same time for the minimum for all the samples to be compared, rather than adjusting the time for slight variations between samples. If the range of times of observed minima for the samples tested exceed 20 min, reliability of the test is suspect.

6.2.2.10 Report the mixture proportions used, including type and amount of calcium sulfate used, mixing regime employed, and the test temperature in the calorimeter.

6.2.3 *Concrete Strength*—Prepare all material according to Practice C192/C192M 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/C78M.

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~~10 °C range) and the same curing temperature (all curing temperatures within ~~4°C~~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 Use the amount of mixing water to produce a flow of 105 to 115 % 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 16—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 17—In the case of strength and heat of hydration measurements maximum performance is normally defined as maximum strength or heat of hydration. In the case drying shrinkage maximum performance is normally defined as minimum drying shrinkage.

NOTE 18—See ~~the~~Appendix X3 ~~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.~~Fit:*

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

$$\text{Performance Level} = a(SO_3)^2 + bSO_3 + c$$

$$\text{performance level} = a(SO_3)^2 + bSO_3 + c$$

where:

a, b, and c = fitting coefficients.

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

NOTE 19—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:

$$\text{Optimum } SO_3 \text{ approximation} = -b/(2\,a)$$

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

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 20—Mathematical and statistics software programs are useful in doing such fits. Calculation of the standard error of estimate can help distinguish between goodness of fit of various fits. Generally the lower the standard error of estimate the better the fit to the measured data. The standard error of estimate, σ_{est}, is defined in Eq 5 below. Examples of calculating the standard error of estimate can be found in the Appendix X3.

$$\sigma_{est} = \sqrt{\frac{\Sigma(P_{measured} - P_{predicted})^2}{n}} \tag{5}$$

$$\sigma_{est} = \sqrt{\frac{\Sigma(P_{measured} - P_{predicted})^2}{n}} \tag{5}$$

where:

$P_{measured}$ = the performance level measured at a particular sulfate level,
$P_{predicted}$ = the performance level predicted from the regression fit at a particular sulfate level, and
n = the number of different sulfate levels tested.

Note 21—The sulfate level for the maximum performance may be the same SO_3 content that one of the tests was conducted at or it may be between two of the SO_3 levels that the tests were 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)

APPENDIXES

(Nonmandatory Information)

X1. PROCESS CONTROL OF SO$_3$ IN HYDRAULIC CEMENT PRODUCTION USING ISOTHERMAL CONDUCTION CALORIMETRY

X1.1 Definition of Terms Used in ~~this~~ Appendix

X1.1.1 *sulfate depletion point, n*—the onset of accelerated calcium aluminate activity that for a portland cement may take place after the main hydration peak, arrow "E" in Fig. 1.

X1.2 Procedure for Determining Target Elapsed Time

X1.2.1 Complete estimation of optimum sulfate using heat of hydration measurements as defined in 6.2.2.

X1.2.1.1 For each different heat of hydration test identify the time elapsed from the maximum thermal power of the main hydration peak, arrow "D" in Fig. 1, to the sulfate depletion point, arrow "E" in Fig. 1 which is "E$_t$-D$_t$~~:~~." This is done using the

thermal power per gram of cement as a function of time data. In cases where the sulfate depletion point cannot be resolved at the sulfate level shown by the heat of hydration testing to be near optimum, the procedure in this appendix does not apply.

Note X1.1—The sulfate depletion point is normally only visible for cements close to and above the optimum SO_3 content. Mathematical methods are useful in locating the sulfate depletion point by finding the local minimum of the heat flow (arrow "E" in Fig. 1) located after the maximum of the main hydration peak (arrow "D" in Fig. 1) and before the heat flow has decreased to 1/3 of the maximum of the main hydration peak. One mathematical method is to use the mathematical derivative of the heat flow curve to more easily determine the time at the different peaks of interest. Experience shows that it is often desirable to low-pass filter the heat flow curve in order to reduce the impact of instrument-specific noise prior to analyzing the curve for the local minimum. As a precaution always visually confirm the software-generated location of the sulfate depletion point prior to reporting. Use of admixtures ~~and/or SCMs~~ or SCMs, or both in this test can have the effect of moving the sulfate depletion point to earlier times, and make it harder to determine.

X1.2.1.2 Using each of the heat of hydration tests determine the E_t-D_t that corresponds to the optimum sulfate level for heat of hydration, E_t-D_t Target. The sulfate level for the maximum heat of hydration may or may be not the SO_3 content that one of the tests was conducted at and interpolation between measured SO_3 contents may be able to provide a better estimate.

X1.3 Verification Tests in Process Control

X1.3.1 Collect representative samples and perform heat of hydration testing without sulfate addition at the same temperature that the optimum sulfate was determined in X1.2.1. Continue tests for 24 h or until the cement hydration has proceeded past the sulfate depletion point, otherwise following Test Method C1702.

X1.3.2 Plot the thermal power per gram of cement as a function of time and identify the time elapsed from the maximum thermal power of the main hydration peak to the sulfate depletion point (E_t-D_t).

X1.3.3 If E_t-D_t differs significantly from the time that corresponds to the mixture at or closest to the optimum SO_3 content in X1.2 then adjustments to the process may be made to bring the E_t-D_t closer to the E_t-D_t Target or an investigation into the cause for the shift from the E_t-D_t Target may be initiated.

Note X1.2—Adjustments to the process frequently involve changing the calcium sulfate dosing to the cement mill, but may also involve changes to milling temperature targets to change the reactivity of calcium sulfate in the cement, changes to the calcium sulfate source added to the mill, adjustments to clinker chemistry, cement fineness, or others.

X2. DISCUSSION OF THE TERM "OPTIMUM"

X2.1 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~~ 23 °C also produces approximately the lowest expansion in water and the lowest contraction in air at that temperature.

X2.2 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.

X2.3 The age for determining the optimum is typically chosen by the manufacturer based on experience with local concrete materials.

X2.4 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.