Designation: C 311 – 00$^{\epsilon 1}$

Standard Test Methods for Sampling and Testing Fly Ash or Natural Pozzolans for Use as a Mineral Admixture in Portland-Cement Concrete[1]

This standard is issued under the fixed designation C 311; 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.

This standard has been approved for use by agencies of the Department of Defense.

ε[1] NOTE—Eq 10 was editorially corrected November 2001.

1. Scope

1.1 These test methods cover procedures for sampling and testing fly ash and raw or calcined pozzolans for use as a mineral admixture in portland-cement concrete.

1.2 The procedures appear in the following order:

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Sampling	7
CHEMICAL ANALYSIS	
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Silicon dioxide, aluminum oxide, iron oxide, calcium oxide, magnesium oxide, sulfur trioxide, sodium oxide and potassium oxide	15
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1.3 The values stated in SI units are to be regarded as the standard. The values given in parentheses are provided for information purposes only.

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.*

1.5 The text of this standard references notes and footnotes that provide explanatory information. These notes and footnotes (excluding those in tables) shall not be considered as requirements of this standard.

2. Referenced Documents

2.1 *ASTM Standards:*

C 33 Specification for Concrete Aggregates[2]

C 109/C 109M Test Method for Compressive Strength of Hydraulic Cement Mortars (Using 2-in. or 50-mm Cube Specimens)[3]

C 114 Test Methods for Chemical Analysis of Hydraulic Cement[3]

C 150 Specification for Portland Cement[3]

C 151 Test Method for Autoclave Expansion of Portland Cement[3]

C 157 Test Method for Length Change of Hardened Hydraulic Cement Mortar and Concrete[2]

C 185 Test Method for Air Content of Hydraulic Cement Mortar[3]

C 188 Test Method for Density of Hydraulic Cement[3]

C 204 Test Method for Fineness of Portland Cement by Air Permeability Apparatus[3]

C 226 Specification for Air-Entraining Additions for Use in the Manufacture of Air-Entraining Portland Cement[3]

C 227 Test Method for Potential Alkali Reactivity of Cement-Aggregate Combinations (Mortar-Bar Method)[2]

C 305 Practice for Mechanical Mixing of Hydraulic Cement Pastes and Mortars of Plastic Consistency[3]

C 430 Test Method for Fineness of Hydraulic Cement by the 45-µm (No. 325) Sieve[3]

C 441 Test Method for Effectiveness of Mineral Admixtures or Ground Blast-Furnace Slag in Preventing Excessive Expansion of Concrete Due to the Alkali-Silica Reaction[2]

C 618 Specification for Coal Fly Ash and Raw or Calcined Natural Pozzolan for Use as a Mineral Admixture in Concrete[2]

C 670 Practice for Preparing Precision and Bias Statements for Test Methods for Construction Materials[2]

C 778 Specification for Standard Sand[3]

C 1012 Test Method for Length Change of Hydraulic-Cement Mortars Exposed to a Sulfate Solution[3]

[1] These test methods are under the jurisdiction of ASTM Committee C09 on Concrete and Concrete Aggregatesand are the direct responsibility of Subcommittee C09.24on Ground Slag and Pozzolonic Admixtures.

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[2] *Annual Book of ASTM Standards*, Vol 04.02.

[3] *Annual Book of ASTM Standards*, Vol 04.01.

C 1157 Performance Specification for Hydraulic Cement[3]
D 4326 Test Method for Major and Minor Elements in Coal and Coke Ash By X-Ray Fluorescence[4]

3. Terminology

3.1 *Definitions of Terms Specific to This Standard:*

3.1.1 *composite sample*—a sample that is constructed by combining equal portions of grab or regular samples.

3.1.2 *established source*—a source for which at least six months of continuous production quality assurance records from a test frequency required for a new source are available, sampled at the source.

3.1.3 *grab sample*—a sample that is taken in a single operation from a conveyor delivering to bulk storage, from bags, or from a bulk shipment. Such a sample may, or may not, reflect the composition or physical properties of a single lot of mineral admixture. This type of sample can be used to characterize small amounts of mineral admixture.

3.1.4 *jobsite or new source*—a source for which less than six months of production records are available, sampled at the source.

3.1.5 *lot*—specific quantity of fly ash or natural pozzolan offered for inspection at any one time. A lot may be one storage bin or the contents of one or more transport units representing mineral admixture drawn from the same storage bin.

3.1.6 *regular sample*—a sample that is constructed by combining equal portions of grab samples that were taken at predetermined times or locations from any single lot of mineral admixture.

4. Significance and Use

4.1 These test methods are used to develop data for comparison with the requirements of Specification C 618. These test methods are based on standardized testing in the laboratory and are not intended to simulate job conditions.

4.1.1 *Strength Activity Index*—The test for strength activity index is used to determine whether a mineral admixture results in an acceptable level of strength development when used with hydraulic cement in concrete. Since the test is performed with mortar, the results may not provide a direct correlation of how the mineral admixture will contribute to strength in concrete.

4.1.2 *Chemical Tests*— The chemical component determinations and the limits placed on each do not predict the performance of a mineral admixture with hydraulic cement in concrete, but collectively help describe composition and uniformity of the mineral admixture.

5. Materials

5.1 *Graded Standard Sand*—The sand used for making test specimens for the activity index with lime or portland cement shall be natural silica sand conforming to the requirements for graded standard sand in Specification C 778.

NOTE 1—*Segregation of Graded Sand*—The graded standard sand should be handled in such a manner as to prevent segregation, since variations in the grading of the sand cause variations in the consistency of the mortar. In emptying bins or sacks, care should be exercised to prevent the formation of mounds of sand or craters in the sand, down the slopes of which the coarser particles will roll. Bins should be of sufficient size to permit these precautions. Devices for drawing the sand from bins by gravity should not be used.

5.2 *Hydrated Lime*— The hydrated lime used in the tests shall be reagent-grade calcium hydroxide, 95 % minimum calculated as $Ca(OH)_2$(Note 2), and have a minimum fineness of 2500 m^2/kg as determined in accordance with Test Method C 204.

NOTE 2—The calcium hydroxide should be protected from exposure to carbon dioxide. Material remaining in an opened container after a test should not be used for subsequent tests.

5.3 *Portland Cement*— The portland cement used in the Strength Activity Index with Portland Cement test shall comply with the requirements of Specification C 150 and have a minimum compressive strength of 35 MPa (5000 psi) at 28 days and total alkalies ($Na_2O + 0.658\ K_2O$) not less than 0.50 % nor more than 0.80 %.

5.3.1 The use of a locally available portland cement in the Strength Activity Index or a project cement that does not meet the requirements of the section on Materials is permitted when the variations from the requirements of the section on Materials are reported and when the use of such portland cement is requested.

6. Sample Type and Size

6.1 Grab samples and regular samples shall have a mass of at least 2 kg (4 lb).

6.2 Grab samples or regular samples taken at prescribed intervals over a period of time (see Table 1), may be combined to form a composite sample representative of the mineral admixture produced during that period of time.

6.3 Composite samples shall have a mass of at least 4 kg (8 lb).

6.4 The sampling shall be done by, or under the direction of, a responsible representative of the purchaser.

7. Sampling Procedure

7.1 The mineral admixture may be sampled by any one of the following methods:

7.1.1 *From Bulk Storage at Point of Discharge or from Rail Cars and Road Tankers.* A sample may be taken by siphon tube during loading or by sampling tube from each loaded car or tanker. If the load is sampled at the point of discharge into the rail car or tanker, the top surface shall be removed to a depth

TABLE 1 Minimum Sampling and Testing Frequency[A]

Test	Sample Type	Jobsite or New Source[B]	Established Source[B]
Moisture content Loss on ignition Fineness	Regular	Daily or each (90 Mg[C] (100 Tons)	Daily or each 360 Mg[C] (400 Tons)
Density and the other tests in Specification C 618, Tables 1 and 2	Composite	Monthly or each 1 800 Mg[C] (2 000 Tons)	Monthly or each 2 900 Mg[C] (3 200 Tons)

[A] It should be noted that the minimum test frequency given in Table 1 is not necessarily the frequency needed for quality control programs on some mineral admixtures.
[B] For definitions, refer to the Terminology section.
[C] Whichever comes first.

[4] *Annual Book of ASTM Standards,* Vol 05.05.

of at least 200 mm (8 in.) before sampling. The sample shall be identified with at least the date and shipment number.

7.1.2 *From Bags in Storage*. The regular sample shall comprise increments of equal size taken by sampling tube from three bags selected at random from one lot of bagged material. The sample shall be identified with date and lot number.

7.1.3 *From Conveyor Delivering to Bulk Storage*. Take one sample of 2 kg (4 lb) or more of the material passing over the conveyor. This may be secured by taking the entire test sample in a single operation, known as the grab sample method, or by combining several equal portions taken at regular intervals, known as the regular sample method. Automatic samplers may be used to obtain samples.

7.2 Samples shall be treated as described in Section 8.

NOTE 3—Some methods of loading or delivery of mineral admixtures, particularly from an airstream or conveyor belt, may create stratification or segregation in the material stream. Sampling techniques must be designed to ensure that the sample is representative of the mineral admixture shipped.

8. Preparation and Storage of Samples

8.1 Prepare composite samples for the tests required in Section 9, by arranging all grab or regular samples into groups covering the period or quantity to be represented by the sample. Take equal portions from each, sufficient to produce a composite sample large enough for the tests required. Mix the composite sample thoroughly.

8.2 Samples shall be stored in clean, airtight containers identified with the source and lot or period of time represented. Untested portions of the sample shall be retained for at least one month after all test results have been reported.

9. Testing Frequency

9.1 *General*—When required, the purchaser shall specify the amount of testing for available alkalies, reactivity with cement alkalies, drying shrinkage, and air-entrainment. Make all other tests on regular or composite samples chosen as specified in Table 1.

CHEMICAL ANALYSIS

10. General

10.1 All apparatus, reagents and techniques shall comply with the requirements of Test Methods C 114.

10.2 *Purity of Water*— Unless otherwise indicated, references to water shall be understood to mean distilled water or water of equal purity.

MOISTURE CONTENT

11. Procedure

11.1 Dry a weighed sample, as received, to constant weight in an oven at 105 to 110°C (221 to 230°F).

12. Calculation

12.1 Calculate the percentage of moisture to the nearest 0.1 %, as follows:

$$\text{Moisture content, \%} = (A/B) \times 100 \tag{1}$$

where:
A = mass loss during drying, and
B = mass as received.

LOSS ON IGNITION

13. Procedure

13.1 Determine loss on ignition in accordance with the procedures outlined in Test Methods C 114, except that the material remaining from the determination of moisture content shall be ignited to constant mass in an uncovered porcelain, not platinum, crucible at 750 ± 50°C (1382 ± 190°F).

14. Calculation

14.1 Calculate the percentage of loss on ignition to the nearest 0.1, as follows:

$$\text{Loss on ignition, \%} = (A/B) \times 100 \tag{2}$$

where:
A = loss in mass between 105 and 750°C (221 and 1382°F),
B = mass of moisture-free sample used.

SILICON DIOXIDE, ALUMINUM OXIDE, IRON OXIDE, CALCIUM OXIDE, MAGNESIUM OXIDE, SULFUR TRIOXIDE, SODIUM OXIDE AND POTASSIUM OXIDE

15. Procedure

15.1 Determine the percentages of these oxides as required in accordance with the applicable sections of Test Methods C 114 for materials having an insoluble residue greater than 1 % (Note 4). Analysts performing sodium oxide and potassium oxide determinations shall observe the precautions outlined in the applicable section of C 1157 (refer to the section on Test Methods). Most pozzolans dissolve completely in lithium borate fluxes.

NOTE 4—Rapid and instrumental methods may be employed similar to those in Test Methods C 114 and D 4326.

AVAILABLE ALKALI

16. Procedure

16.1 Weigh 5.0 g of the sample and 2.0 g of hydrated lime on a piece of weighing paper, carefully mix using a metal spatula, and transfer to a small plastic vial of approximately 25-mL capacity. Add 10.0 mL of water to this mixture, seal the vial by securing the cap or lid to the vial with tape (Note 5), blend by shaking until the mixture is uniform, and store at 38 ± 2°C.

NOTE 5—To ensure that moisture loss from the paste does not occur, place the sealed vial in a sealable container (such as a small sample or mason jar), add sufficient water to cover the bottom of the container, and seal.

16.2 Open the vial at the age of 28 days and transfer the contents to a 250-mL casserole. Break up and grind the cake with a pestle, adding a small amount of water, if necessary, so that a uniform slurry containing no lumps is obtained (Note 6). Add sufficient water to make the total volume 200 mL. Let stand 1 h at room temperature with frequent stirring. Filter through a medium-textured filter paper onto a 500-mL volumetric flask. Wash thoroughly with hot water (eight to ten times).

NOTE 6—At times it may be necessary to break the vial and peel off the plastic from the solid cake. In such cases, care should be exercised to avoid the loss of material and to remove all solid material from the fragments of the vial. If the cake is too hard to break up and grind in the casserole, a mortar should be used.

16.3 Neutralize the filtrate with dilute HCl (1 + 3), using 1 to 2 drops of phenolphthalein solution as the indicator. Add exactly 5 mL of dilute HCl (1 + 3) in excess. Cool the solution to room temperature and fill the flask to the mark with distilled water. Determine the amount of sodium and potassium oxides in the solution using the flame photometric procedure, described in Test Methods C 114, except that the standard solutions shall be made up to contain 8 mL of calcium chloride ($CaCl_2$) stock solution per litre of standard solution, and the solution as prepared shall be used in place of the solution of cement.

NOTE 7—The standard solutions made up with 8 mL of calcium chloride ($CaCl_2$) stock solution contain the equivalent of 504 ppm of CaO. Tests have shown that this amount closely approximates the amount of calcium dissolved in the test solution.

17. Calculation and Report

17.1 Calculate the results as weight percent of the original sample material. Report as equivalent percentage of sodium oxide (Na_2O), calculated as follows:

$$\text{Equivalent } Na_2O, \% = Na_2O, \% + 0.658 \times K_2O, \% \quad (3)$$

PHYSICAL TESTS

DENSITY

18. Procedure

18.1 Determine the density of the sample in accordance with the procedure described in Test Method C 188, except use about 50 g of mineral admixture instead of approximately 64 g of cement as recommended in Test Method C 188.

FINENESS, AMOUNT RETAINED WHEN WET-SIEVED ON A 45-µm (NO. 325) SIEVE

19. Procedure

19.1 Determine the amount of the sample retained when wet-sieved on a 45-µm (No. 325) sieve, in accordance with Test Method C 430, with the following exceptions.

19.1.1 Calibrate the 45-µm (No. 325) sieve using a cement standard (SRM 114).[5] Calculate the sieve correction factors as follows:

$$CF = std - obs \quad (4)$$

where:

CF = the sieve correction factor, %, (include a negative sign when appropriate),
std = the certified residue value for the SRM, %, and
obs = the observed residue value for the SRM, %.

19.1.2 Calculate the fineness of the mineral admixture to the nearest 0.1 % as follows:

$$R_C = R_S + CF \quad (5)$$

where:

R_C = corrected sieve residue, %,
R_S = observed residue for the test sample, %, and
CF = the sieve correction factor, %.

NOTE 8—Test Method C 430 has been adopted for testing fly ash fineness. However, certain requirements, such as cleaning of sieves and interpretation of the test results, are sometimes not appropriate for fly ashes. The C 311 task group is currently evaluating the use of SRM fly ashes for performing sieve calibrations.

INCREASE OF DRYING SHRINKAGE OF MORTAR BARS

20. Test Specimen

20.1 Prepare test specimens in accordance with the procedures described in Test Method C 157, except mold three mortar bars from both the control mix and the test mix using the following proportions:

	Control Mix	Test Mix
Portland cement, g	500	500
Mineral admixture, g	None	125
Graded standard sand, g	1375	1250
Water	sufficient to produce a flow of 100 to 115 %	

21. Procedure

21.1 Cure and measure the test specimens in accordance with Test Method C 157, except that the moist-curing period (including the period in the molds) shall be 7 days, and the comparator reading at the age of 24 ± ½ h shall be omitted. Immediately after taking the comparator reading at the end of the 7-day moist-curing period, store the specimens in accordance with Test Method C 157, and after 28 days of air storage, take a comparator reading for the specimens in accordance with Test Method C 157.

22. Calculation and Report

22.1 Calculate the increase in drying shrinkage of the mortar bars, S_i, as follows:

$$S_i = S_t - S_c \quad (6)$$

where:

S_t = average drying shrinkage of the test specimens calculated as follows, and
S_c = average drying shrinkage of the control specimens calculated as follows:

$$S = \frac{[\text{initial CRD} - \text{CRD}] \times 100}{G} \quad (7)$$

where:

S = drying shrinkage of test or control specimens, %,
initial CRD = difference between the comparator reading of the specimen and the reference bar at 7 days of moist curing,
CRD = difference between the comparator reading of the specimen and the reference bar at 28 days of drying, and

[5] Available from the National Institute of Standards and Technology (NIST), Gaithersburg, MD 20899.

G = the gage length of the specimens 250 mm (10 in.).

22.2 Report the results to the nearest 0.01. If the average drying shrinkage of the control specimens is larger than the average drying shrinkage of the test specimens, prefix a minus sign to the increase of drying shrinkage of mortar bars reported.

SOUNDNESS

23. Procedure

23.1 Conduct the soundness test in accordance with Test Method C 151, except that the specimens shall be molded from a paste composed of 25 parts by weight of mineral admixture and 100 parts by weight of a portland cement conforming to Specification C 150.

AIR-ENTRAINMENT OF MORTAR

24. Procedure

24.1 Using portland cement conforming to the requirements for Type I or Type II of Specification C 150, prepare a test mixture in accordance with Test Method C 185, using the following proportions:

	Test Mix
Portland cement, g	300
Mineral admixture	75
20–30 standard sand, g	1125
Water, mL, sufficient to give a flow of 80 to 95	Y
Neutralized Vinsol[6] resin solution, mL,[A] sufficient to produce an air content of 18 ± 3 %	Z

[A] The amount of Vinsol resin solution used shall be considered as part of the mixing water.

24.2 The neutralized Vinsol resin solution used in this section on Air-Entrainment of Mortar shall be either a commercial neutralized Vinsol resin solution or a neutralized Vinsol resin solution prepared in accordance with Specification C 226. If it is necessary to dilute either of these solutions, use distilled or demineralized water. (Note 9.)

NOTE 9—Dissolved minerals in drinking water may precipitate Vinsol resin solutions and greatly diminish its air-entraining characteristics.

24.3 Prepare two test mixtures with sufficient neutralized Vinsol[6] resin to produce an air content of 15 to 18 % in the first mix and 18 to 21 % in the second mix. Then, determine by interpolation the amount of Vinsol resin, expressed as weight percent of the cement, required to produce an air content of 18 %.

25. Calculation

25.1 Calculate the air content of the test mixtures as follows:

$$\text{Air content, volume } \% = 100[1-(W_a/W_c)] \quad (8)$$

$$W_a = W/400 \quad (9)$$

$$W_c = \frac{300+1125+75+(300 \times P \times 0.01)}{\left[\left(\frac{300}{3.15}\right)+\left(\frac{1125}{2.65}\right)+\left(\frac{75}{D}\right)+\left(\frac{300 \times P \times 0.01}{1}\right)\right]} \quad (10)$$

where:

W_a = actual weight per unit of volume of mortar as determined by Test Method C 185, g/mL,
W = weight of the specified 400 mL of mortar (see Test Method C 185), g,
W_c = theoretical weight per unit of volume, calculated on an air-free basis and using the values for density and quantities of the materials in the mix, g/mL,
P = percentage of mixing water plus Vinsol resin solution based on weight of cement, and
D = density of mineral admixture in the mixture, Mg/m^3.

STRENGTH ACTIVITY INDEX WITH PORTLAND CEMENT

26. Specimens

26.1 Mold the specimens from a control mixture and from a test mixture in accordance with Test Method C 109/C 109M. The portland cement used in the Strength Activity Index test shall comply with the requirements of Specification C 150 and with the alkali and strength limits given in the section on Materials. In the test mixture, replace 20 % of the mass of the amount of cement used in the control mixture by the same mass of the test sample. Make six-cube batches as follows:

26.1.1 Control Mixture:

500 g of portland cement
1375 g of graded standard sand
242 mL of water

26.1.2 Test Mixture:

400 g of portland cement
100 g of test sample
1375 g of graded standard sand
mL of water required for flow ±5 of control mixture

26.2 *Number of Specimens*:

26.2.1 Since Specification C 618 specifies that "meeting the 7 day or 28 day Strength Activity Index will indicate specification compliance" only one age might be required. At the option of the producer or the user after preparing six-cube batches, only three cubes of control and test mixtures need to be molded for either 7 or 28 day testing.

27. Storage of Specimens

27.1 After molding, place the specimens and molds (on the base plates) in the moist room or closet at 23.0 ± 2.0°C (73.4 ± 3°F) for 20 to 24 h. While in the moist room or closet, protect the surface from dripping water. Remove the molds from the moist room or closet and remove the cubes from the molds. Place and store the cubes in saturated lime water as specified in Test Method C 109/C 109M.

NOTE 10—Take care to ensure against zones of stratification or pockets of variation in temperature in the curing chamber.

28. Compressive Strength Test

28.1 Determine the compressive strength, as specified in

[6] Available from Hercules, Inc., 917 King St., Wilmington, DE 19899.

Test Method C 109/C 109M, of three specimens of the control mixture and three specimens of the test mixture at ages of 7 days, or 28 days, or both, depending upon how many specimens were molded as prescribed in the section on Number of Specimens.

29. Calculation

29.1 Calculate the strength activity index with portland cement as follows:

Strength activity index with portland cement =

$$(A/B) \times 100 \quad (11)$$

where:

A = average compressive strength of test mixture cubes, MPa (psi), and

B = average compressive strength of control mix cubes, MPa (psi).

WATER REQUIREMENT

30. Calculation

30.1 Calculate the water requirement for the Strength Activity Index with Portland Cement as follows:

Water requirement, percentage of control =

$$(Y/242 \times 100) \quad (12)$$

where:

Y = water required for the test mixture to be ±5 of control flow.

EFFECTIVENESS OF MINERAL ADMIXTURE IN CONTROLLING ALKALI SILICA-REACTIONS (SEE Appendix X1)

31. Procedure

31.1 Determine expansion of mortar made with a mineral admixture and a test cement as a percent of expansion of mortar made with low alkali cement in accordance with Test Method C 441 as modified in the following paragraphs:

31.1.1 *Control Mixture*— The control mixture will be made as required in Test Method C 441 except that the control cement shall have an alkali content (as equivalent Na_2O) less than 0.60 % (Note 11).

NOTE 11—Generally, the control cement should have an alkali content as equivalent Na_2O between 0.50 and 0.60 %. However, lower alkali control cement may be used, if desired, to demonstrate equivalence.

31.1.2 *Test Mixture Using Mineral Admixture*—The combined quantity of cement plus mineral admixture shall total 400 g (see Appendix X1). Use 900 g of borosilicate glass[7] aggregate and sufficient mixing water to produce a flow between 100 and 115 % as determined in accordance with Test Method C 109/C 109M. The cement used in the test mixture shall have an alkali content greater than that of the cement in the control mixture (Note 12).

NOTE 12—Generally, this test cement will have an alkali content equal to or higher than that used in the job.

31.1.3 Store and measure specimens as required in Test Method C 227. Measure length of specimens at ages of 1 and 14 days.

32. Report

32.1 Include in the report for each test mixture comparison made:

32.1.1 The 14 day expansion of the test mixture as a percent of the control mixture at that age,

32.1.2 The identification and chemical analysis of the mineral admixture,

32.1.3 The mass percentage of mineral admixture based on the total mass of cement plus mineral admixture in the test mixture, and

32.1.4 The alkali content of control and test mixture cements as equivalent alkali (Na_2O + 0.658 K_2O).

EFFECTIVENESS OF MINERAL ADMIXTURES IN CONTRIBUTING TO SULFATE RESISTANCE

33. Procedure

33.1 Compare the length change of mortar bars with absolute expansion limits or compare the length change of mortar bars made with a control cement with the length change of mortar bars made with fly ash or natural pozzolan and a test cement, in accordance with Test Method C 1012, as modified in the following paragraphs. Results shall be evaluated using absolute limits (Procedure A) or the relative expansion limits (Procedure B) in Specification C 618, Table 2A.

33.2 *Control Mixture for Procedure A*—A control mixture is not mandatory since performance measured using Procedure A is based on maximum expansion of the test mixture. If a control mixture is made for Procedure A then proportion it as required in Test Method C 1012 using a cement meeting the requirements of Specification C 150, Type II or Type V.

33.3 *Test Mixture Using Fly Ash or Natural Pozzolan for Procedure A*—Make the combined quantity of cement plus fly ash or natural pozzolan, by mass, the same as the total cement content of the control mixture described in the Making Mortars section of Test Method C 1012. The proportion of fly ash or natural pozzolan may be varied using from 15 % to 50 %, by mass, of the total cement plus fly ash or natural pozzolan. Any type of portland cement may be used to prepare test mixtures.

33.4 *Control Mixture for Procedure B*—Make the control mixture for Procedure B as required in Test Method C 1012 with the cement that is proposed for use in the project or a cement that through performance or definition (Specification C 150, Section 1) is expected to give satisfactory results (or a cement for which the contribution to sulfate resistance is known and is satisfactory).

NOTE 13—The control cement should be chosen to give sulfate resistance for the expected level of sulfate exposure. Experience has shown that Type II cements are often used for moderate levels of exposure. Type V cement is commonly used for severe exposures (see X2.2 of Appendix X2).

33.5 *Test Mixture for Using Fly Ash or Natural Pozzolan for Procedure B*—Make the combined quantity of cement plus fly

[7] Pyrex brand glass No. 7740 is available from Corning Glass Works, Corning, NY.

ash or natural pozzolan, by mass, the same as the total cement used in the control mixture. The proportion of fly ash or natural pozzolan may be varied from 15 % to 50 % of the total cement plus fly ash or natural pozzolan by mass. Any type of portland cement may be used to prepare test mixtures.

33.6 For either Procedure A or B, store and measure specimens as required in Test Method C 1012 for at least 6 months.

NOTE 14—Evaluation of the sulfate resistance of cementitious materials for use in certain situations or critical structures may require longer periods of storage and additional length measurements. Refer to Test Method C 1012 for guidance on this matter.

34. Report

34.1 In addition to the information required in Test Method C 1012, report the following information for each mixture comparison made:

34.2 For Procedure A, the age and expansion as a specific amount,

34.3 For Procedure B, the age and expansion as a percent of the control mixture at that age,

34.4 The identification and chemical analysis including C_3A content of the cements used in both the control and test mixtures,

34.5 The identification and chemical analysis of the fly ash or natural pozzolan used in the test mixtures,

34.6 The mass percentages of the fly ash or natural pozzolan based on the total mass of cement plus fly ash or natural pozzolan in the test mixture.

PRECISION AND BIAS

35. Precision and Bias

35.1 *Strength Activity Index Test*:

35.1.1 Precision was determined from two interlaboratory studies involving two Class C fly ashes, two Class F fly ashes, and one Class N pozzolan. Participating laboratories numbered 12 and 7 in the two studies.[8]

35.1.2 The single-operator standard deviation for the Strength Activity Index test has been found to be 3.7 % (1s). This does not appear to vary either with material or with test age, over the range of 7 to 28 days. Therefore, results of two properly conducted tests by the same operator on the same material should not differ by more than 10.5 % (d2s) of the average of the two results.

35.1.3 The multilaboratory standard deviation for the Strength Activity Index test has been found to be 4.5 % (1s). This does not appear to vary either with material or with test age, over the range of 7 to 28 days. Therefore, results of two properly conducted tests in different laboratories on the same material should not differ by more than 12.7 % (d2s) of the average of the two results.

35.1.4 Since there is no accepted reference material suitable for determining the bias for this procedure, no statement on bias is being made.

35.2 *Chemical Analysis*:

35.2.1 Precision and bias estimates for the test methods in C 114 when applied to the analysis of pozzolans were calculated from an interlaboratory study involving 7 laboratories each analysing 4 NIST SRM fly ashes. A research report describing the results of this study is available from ASTM.[9]

35.2.2 *Precision*—Within-laboratory (W/L) and between-laboratory (B/L) estimates of standard deviation and estimates of maximum differences expected between duplicate determinations in 95 % of comparisons[10] are summarized in Table 2.

35.2.3 *Bias*—Statistically significant bias was found in the determination of CaO and MgO. CaO determinations averaged 0.46 % higher than the value certified for the SRM. MgO determinations averaged 0.19 % higher than the value certified for the SRM.

TABLE 2 Within and Between Lab Precision Estimates for C 114 Methods Applied to the Analysis of Fly Ash[A]

Oxide	1s		d2s	
	W/L	B/L	W/L	B/L
SiO_2	0.62	0.86	1.75	2.45
Al_2O_3	1.46	2.29	4.12	6.48
Fe_2O_3				
<2 %	0.12	0.11	0.34	0.31
≥2 %	0.12	0.25	0.34	0.71
CaO	0.63	0.74	1.79	2.09
MgO	0.20	0.21	0.55	0.61
SO_3	0.10	0.16	0.28	0.45
Moisture	0.05	0.06	0.15	0.17
LOI	0.09	0.12	0.25	0.35

[A]The four values for SO_3 were editorially corrected to reflect updated research reports associated with this standard.

36. Keywords

36.1 fly ash; natural pozzolan

[8] A research report summarizing the statistical analysis is available from ASTM Headquarters, 100 Barr Harbor Drive, West Conshohocken, PA 19428. Request RR: C09-1001.

[9] A research report is available from ASTM Headquarters, 100 Barr Harbor Drive, West Conshohocken, PA 19428. Request RR: C09-1008.

[10] These values represent, respectively, the (1s) and (d2s) limits as described in Practice C 670.

APPENDIXES

(Nonmandatory Information)

X1. COMMENTARY ON SECTION 31

X1.1 Test procedures in Section 31 are designed to determine the effectiveness of fly ash, or natural pozzolan, in preventing excessive expansion resulting from a reaction between certain aggregates and alkalies in portland-cement mixtures. Tests are made in accordance with Test Method C 441 using:

X1.1.1 400 g of portland cement or a combined total of cement plus fly ash or natural pozzolan of 400 g,

X1.1.2 900 g of borosilicate glass,[7] and

X1.1.3 Sufficient water to obtain a flow of 100 to 115 %.

X1.2 The control mixture is made with a low-alkali portland cement. The test mixture, made of a test percentage of the fly ash or natural pozzolan, can be made with the same cement or any other cement having an equivalent alkali content greater than 0.60 %.

X1.3 *Interpretation of Results*—The fly ash or natural pozzolan should be considered "effective" when used at percentages equal to or greater than the percentages used in the test mixture with cements having alkali contents that do not exceed by more than 0.05 percentage points the alkali content of the cement used in the test mixture.

X1.4 *Selection of the Percentage of Fly Ash or Natural Pozzolan*—It may be necessary to make test mixtures with several percentages of fly ash or natural pozzolan to determine the minimum amount necessary to be considered "effective" in reducing expansion to the level produced by the "control" low-alkali cement. The minimum amount of fly ash or natural pozzolan should be 15 % by weight of cementitious material.

X1.5 *Selection of the Alkali Content of the Cement Used in the Test Mixture*—In some instances it may not be necessary to demonstrate that the fly ash or natural pozzolan will reduce expansion, but rather that it will not increase expansion. In this instance the test and control mixtures should be made with the same low alkali cement used in the control mixture to better delineate any increase in expansion. The fly ash or natural pozzolan percentage used may need to be sufficiently high to demonstrate that the percentage exceeds the pessimum if such pessimum exists for the combination.

X1.6 *Selection of the Low Alkali Control Cement*—The test procedure in this test method and requirements in Specification C 618 are designed to measure the relative effectiveness of the fly ash or natural pozzolan in reducing expansion due to alkali-silica reactions. The effectiveness is a function of both the alkali content of the cement used in the test mixture and the percentage of fly ash or natural pozzolan. The higher the alkali content of the test mixture cement and the lower the percentage of fly ash or natural pozzolan used, the more effective the fly ash or natural pozzolan. This test procedure and the 100 % criterion of Specification C 618 can be used as a guide to enhance field performance of job mixtures if it is assumed that use of the low alkali control cement will prevent excessive expansion in service with materials proposed for use in the job. This is the situation when the aggregates proposed for use contain rapidly reactive constituents but do not exceed 0.05 % expansion in three months or 0.10 % expansion in six months when tested with the control cement in Test Method C 227. In such instances the control cement used in this test method should have an alkali content as required to control the expansive reactions of the aggregates being used, for example in the 0.50 to 0.60 % range. See the appendix to Specification C 33.

X1.6.1 Certain reactive aggregates have been identified which produce deleterious expansion after many years of service when used with cements with equivalent alkali contents well below 0.60 %. Often these reactive aggregates do not produce expansions in Test Method C 227 exceeding those discussed in the appendix of Specification C 33 until a much later age, perhaps one or two years. In such instances the use of a control cement with an alkali content significantly less than 0.60 % is appropriate.

X2. COMMENTARY ON SECTION 33

X2.1 The test procedures in Section 33 are designed to evaluate the performance of particular fly ashes or natural pozzolans in contributing to the resistance or durability of concrete in a sulfate environment. Tests are made on mortars in accordance with Test Method C 1012.

X2.2 The control mixture should be made with a cement that by performance or definition (Specification C 150, Section 1.1) is expected to give satisfactory results for the anticipated level of sulfate exposure. The American Concrete Institute Guide to Durable Concrete (ACI 201.2R) recommends Type II cement when the sulfate content, expressed as SO_4, is 0.10 to 0.20 percent in soils or 150 to 1500 ppm in waters. The American Concrete Institute Guide to Durable Concrete (ACI 201.2R) recommends Type V cements when the sulfate content, expressed as SO_4, is 0.20 to 2.0 % in soils, or 1500 to 10 000 ppm in waters. The test mixtures for both procedures may be made with varying percentages of fly ash or natural pozzolan. Test mixtures can use cements with equal or higher or lower C_3A contents that might not have given satisfactory results when used alone.

X2.3 *Interpretation of Results*—The absolute expansion limits used in Procedure A make it difficult to ascertain how the addition of fly ash or natural pozzolan influences sulfate resistance. However, as referenced in Test Method C 1012, research has indicated that mortars meeting the expansion criteria will perform adequately. Under Procedure B, the fly ash or natural pozzolan will be considered to be able to contribute to sulfate resistance if the expansion of the test mixture does not exceed that of the control mixture at an exposure time of at least 6 months. It is recommended that the proportion of fly ash or natural pozzolan used in the test mixture be the same one proposed for use in the project ±2 %, and that the C_3A content of the project cement be equal to, or less than, that which was used in the test mixtures. See Appendix X1, Technical Background, Test Method C 1012.

X2.4 *Selection of the Percentage of Fly Ash or Natural Pozzolan*—It may be necessary to make test mixtures with several percentages of fly ash or natural pozzolan to determine the amount necessary to obtain adequate resistance to different concentrations of sulfates. Percentages used in a project should be within 2 % of those that are successful in the test mixtures or should be between two percentages that are successful.