Designation: C 311 – 00

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.

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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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]

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]

[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.

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

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 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

TABLE 1 Minimum Sampling and Testing Frequency[A]

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

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:

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

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:

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

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, %.

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

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
- 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.