



Designation: C311/C311M – 16

Standard Test Methods for Sampling and Testing Fly Ash or Natural Pozzolans for Use in Portland-Cement Concrete¹

This standard is issued under the fixed designation C311/C311M; 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 U.S. 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 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	15
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1.3 The values stated in either SI units or inch-pound units are to be regarded separately as standard. The values stated in each system may not be exact equivalents; therefore, each system shall be used independently of the other. Combining values from the two systems may result in non-conformance with the standard.

¹ These test methods are under the jurisdiction of ASTM Committee C09 on Concrete and Concrete Aggregates and are the direct responsibility of Subcommittee C09.24 on Supplementary Cementitious Materials.

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NOTE 1—Sieve size is identified by its standard designation in Specification E11. The alternative designation given in parentheses is for information only and does not represent a different standard sieve size.

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.

1.6 *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
- C33/C33M Specification for Concrete Aggregates
 - 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
 - C125 Terminology Relating to Concrete and Concrete Aggregates
 - C150/C150M Specification for Portland Cement
 - C151/C151M Test Method for Autoclave Expansion of Hydraulic Cement
 - C157/C157M Test Method for Length Change of Hardened Hydraulic-Cement Mortar and Concrete
 - C185 Test Method for Air Content of Hydraulic Cement Mortar

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

C188 Test Method for Density of Hydraulic Cement
C204 Test Methods for Fineness of Hydraulic Cement by Air-Permeability Apparatus
C219 Terminology Relating to Hydraulic Cement
C226 Specification for Air-Entraining Additions for Use in the Manufacture of Air-Entraining Hydraulic Cement
C227 Test Method for Potential Alkali Reactivity of Cement-Aggregate Combinations (Mortar-Bar Method)
C430 Test Method for Fineness of Hydraulic Cement by the 45- μm (No. 325) Sieve
C441/C441M Test Method for Effectiveness of Pozzolans or Ground Blast-Furnace Slag in Preventing Excessive Expansion of Concrete Due to the Alkali-Silica Reaction
C604 Test Method for True Specific Gravity of Refractory Materials by Gas-Comparison Pycnometer
C618 Specification for Coal Fly Ash and Raw or Calcined Natural Pozzolan for Use in Concrete
C670 Practice for Preparing Precision and Bias Statements for Test Methods for Construction Materials
C778 Specification for Standard Sand
C1012/C1012M Test Method for Length Change of Hydraulic-Cement Mortars Exposed to a Sulfate Solution
C1437 Test Method for Flow of Hydraulic Cement Mortar
C1697 Specification for Blended Supplementary Cementitious Materials
D1426 Test Methods for Ammonia Nitrogen In Water
D4326 Test Method for Major and Minor Elements in Coal and Coke Ash By X-Ray Fluorescence
E11 Specification for Woven Wire Test Sieve Cloth and Test Sieves
 2.2 *ACI Document*.³
ACI 201.2R Guide to Durable Concrete

3. Terminology

3.1 Definitions:

3.1.1 For definitions of terms used in this test method, refer to Terminology **C125** and Terminology **C219**.

3.2 Definitions of Terms Specific to This Standard:

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

3.2.2 *established source, n*—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.

3.2.3 *grab sample, n*—a sample that is taken in a single operation from a conveyor delivering to bulk storage, from bags, or from a bulk shipment.

3.2.3.1 *Discussion*—A grab sample may or may not reflect the composition or physical properties of a single lot of fly ash or natural pozzolan. This type of sample can be used to characterize small amounts of material.

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

³ Available from American Concrete Institute (ACI), P.O. Box 9094, Farmington Hills, MI 48333-9094, <http://www.aci-int.org>.

3.2.5 *regular sample, n*—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 material.

4. Significance and Use

4.1 These test methods are used to develop data for comparison with the requirements of Specification **C618** or Specification **C1697**. 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 fly ash or natural pozzolan 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 fly ash or natural pozzolan 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 fly ash or natural pozzolan with hydraulic cement in concrete, but collectively help describe composition and uniformity of the material.

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 **C778**.

NOTE 2—*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 $\text{Ca}(\text{OH})_2$ (Note 3), and have a minimum fineness of 2500 m^2/kg as determined in accordance with Test Method **C204**.

NOTE 3—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 **C150/C150M** and have a minimum compressive strength of 35 MPa (5000 psi) at 28 days and total alkalis ($\text{Na}_2\text{O} + 0.658 \text{K}_2\text{O}$) 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 fly ash or natural pozzolan 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 fly ash or natural pozzolan 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 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 4—Some methods of loading or delivery of fly ash or natural pozzolan, 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 material 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

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	Composite	(100 Tons)	(400 Tons)
Density and the other tests in Specification		Monthly or each	Monthly or each
C618, Tables 1 and 2		1 800 Mg ^C (2 000 Tons)	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 fly ash or natural pozzolans.

^B For definitions, refer to the Terminology section.

^C Whichever comes first.

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 alkalis, reactivity with cement alkalis, 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 **C114**.

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 mass in an oven at 110 ± 5 °C [230 ± 10 °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 **C114**, 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.

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 110 and 750 °C,

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 **C114** for materials having an insoluble residue greater than 1 % (**Note 5**). Analysts performing sodium oxide and potassium oxide determinations shall observe the precautions outlined in the applicable section of Test Methods **C114**. Most pozzolans dissolve completely in lithium borate fluxes.

NOTE 5—Rapid and instrumental methods may be employed similar to those in Test Methods **C114** and **D4326**.

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 6**), blend by shaking until the mixture is uniform, and store at 38 ± 2 °C.

NOTE 6—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 7**). 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 7—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 **C114**, 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 8—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:

$$\text{Equivalent Na}_2\text{O, \%} = \text{Na}_2\text{O, \%} + 0.658 \times \text{K}_2\text{O, \%} \quad (3)$$

AMMONIA

18. Procedure

18.1 Weigh 1.00 g of the fly ash sample into a 125-mL Erlenmeyer flask. Add 100 mL of ammonia-free water. Place a neoprene rubber stopper on the flask and swirl the contents to thoroughly mix the sample and the water.

18.2 Filter the mixture using a medium-textured filter paper and save the filtrate for the ammonia determination.

18.3 Determine the concentration of ammonia in the filtrate in accordance with the procedures outlined in Test Methods **D1426**, Method A—Direct Nesslerization or Method B—Selective Ion Electrode.

18.4 Calculate the ammonia concentration of the fly ash as follows:

$$\text{Ammonia, mg/kg} = N_w \times V_w / W_{fa} \quad (4)$$

where:

N_w = ammonia concentration of the water extract determined by Test Methods **D1426**, mg/L,

V_w = volume of water used for extracting ammonia from the fly ash sample, mL, and

W_{fa} = mass of fly ash sample used in the test, g.

PHYSICAL TESTS

DENSITY

19. Procedure

19.1 Determine the density of the sample in accordance with the procedure described in Test Method **C188** or Test Method **C604** as modified below.

19.2 For Test Method **C188**, follow the procedure except use a quantity of fly ash or natural pozzolan weighed to the nearest 0.05 g, of about 50 g.

19.3 For Test Method **C604** use an amount of material that is appropriate for the instrument.

19.3.1 Determine the density of the material as received. Do not prepare sample as described in Section 6 of Test Method **C604**.

19.4 In the final report of the density value, indicate which test method was used in measuring density.

20. Procedure

20.1 Determine the amount of the sample retained when wet-sieved on a 45- μ m (No. 325) sieve, in accordance with Test Method C430, with the following exceptions.

20.1.1 Calibrate the 45- μ m (No. 325) sieve using a cement standard (SRM 114). Calculate the sieve correction factors as follows:

$$CF = std - obs \quad (5)$$

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, %.

20.1.2 Calculate the fineness of the fly ash or natural pozzolan to the nearest 0.1 % as follows:

$$R_c = R_s + CF \quad (6)$$

where:

R_c = corrected sieve residue, %, <https://standards.iteh.ai/>

R_s = observed residue for the test sample, %, and

CF = the sieve correction factor, %.

If the residue retained for the test sample is equal to zero ($R_s = 0$), then the sieve correction factor shall not be added to the test result to calculate the corrected sieve residue. In such cases, the corrected fineness shall be reported as zero.

NOTE 9—Test Method C430 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.

20.2 Numerical examples for calibrating a fineness sieve and calculating the corrected fineness.

20.2.1 Calibrating a fineness sieve (NIST standard reference material SRM 114p was used in this example):

Certified residue retained on a 45 - μ m sieve
= 8.24 % (obtained from standard certificate)

Measured residue retained on a 45 - μ m sieve
= 7.12 % (measured in the laboratory)

Correction factor (CF)
= standard value (std) - observed value (obs)
= 8.24 - 7.12 = 1.12 %

20.2.2 Calculating a corrected fineness value for a calibrated sieve (fly ash A was used in this example):

Amount of fly ash A retained on the sieve: R_s
= 15.2 % (expressed as a % of sample mass)

Corrected sieve residue for fly ash A: R_c
= $R_s + CF$
= 15.2 + 1.12
= 16.3 %

21. Test Specimen

21.1 Prepare test specimens in accordance with the procedures described in Test Method C157/C157M, 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
Fly ash or natural pozzolan, g	None	125
Graded standard sand, g	1375	1250
Water	sufficient to produce a flow of 100 to 115 %	

22. Procedure

22.1 Cure and measure the test specimens in accordance with Test Method C157/C157M, except that the moist-curing period (including the period in the molds) shall be 7 days, and the comparator reading at the age of $24 \pm \frac{1}{2}$ 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 C157/C157M, and after 28 days of air storage, take a comparator reading for the specimens in accordance with Test Method C157/C157M.

23. Calculation and Report

23.1 Calculate the increase in drying shrinkage of the mortar bars, S_p , as follows:

$$S_p = S_t - S_c \quad (7)$$

where: <https://standards.iteh.ai/>

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:

$$S = \frac{[\text{initial CRD} - \text{CRD}] \times 100}{G} \quad (8)$$

where:

S = drying shrinkage of test or control specimens, %, <https://standards.iteh.ai/>

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.).

23.2 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

24. Procedure

24.1 Conduct the soundness test in accordance with Test Method **C151/C151M**, except the specimens shall be molded from a paste composed of water, the fly ash or natural pozzolan being tested, and portland cement conforming to Specification **C150/C150M**. The quantity of fly ash or natural pozzolan shall be 20 % by mass of the total cementitious material.

AIR-ENTRAINMENT OF MORTAR

25. Procedure

25.1 Using portland cement conforming to the requirements for Type I or Type II of Specification **C150/C150M**, prepare a test mixture in accordance with Test Method **C185**, using the following proportions:

	Test Mix
Portland cement, g	300
Fly ash or natural pozzolan	75
20–30 standard sand, g	1125
Water, mL, sufficient to give a flow of 80 to 95	Y
Neutralized Vinsol 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.

25.2 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 **C226**. If it is necessary to dilute either of these solutions, use distilled or demineralized water. (**Note 10**.)

NOTE 10—Dissolved minerals in drinking water may precipitate Vinsol resin solutions and greatly diminish its air-entraining characteristics.

25.3 Prepare two test mixtures with sufficient neutralized Vinsol 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 mass percent of the cement, required to produce an air content of 18 %.

26. Calculation

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

$$\text{Air content, volume \%} = 100[1 - (W_a/W_c)] \quad (9)$$

$$W_a = W/400 \quad (10)$$

$$W_c = \frac{300 + 1125 + 75 + (300 \times P \times 0.01)}{\left[\left(\frac{300}{3.15} \right) + \left(\frac{1125}{2.65} \right) + \left(\frac{75}{D} \right) + \left(\frac{300 \times P \times 0.01}{1} \right) \right]} \quad (11)$$

where:

W_a = actual mass per unit of volume of mortar as determined by Test Method **C185**, g/mL,

W = mass of the specified 400 mL of mortar (see Test Method **C185**), g,

W_c = theoretical mass 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 mass of cement, and

D = density of fly ash or natural pozzolan in the mixture, Mg/m³.

STRENGTH ACTIVITY INDEX WITH PORTLAND CEMENT

27. Specimens

27.1 Mold the specimens from a control mixture and from a test mixture in accordance with Test Method **C109/C109M**. The portland cement used in the Strength Activity Index test shall comply with the requirements of Specification **C150/C150M** 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:

27.1.1 Control Mixture:

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

27.1.2 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

27.2 *Number of Specimens:*

27.2.1 Since Specification **C618** 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.

28. Storage of Specimens

28.1 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 **C109/C109M**.

NOTE 11—Take care to ensure against zones of stratification or pockets of variation in temperature in the curing chamber.

29. Compressive Strength Test

29.1 Determine the compressive strength, as specified in Test Method **C109/C109M**, of three specimens of the control