



Designation: C 226 – 9602

Standard Specification for Air-Entraining Additions for Use in the Manufacture of Air- Entraining Hydraulic Cement¹

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

1. Scope

1.1 This specification covers the requirements and methods for establishing the suitability of a material for use as an air-entraining addition to be interground with the clinker in the manufacture of air-entraining hydraulic cement conforming to Specifications C 150, C 595, and C 1157.

1.2 The values stated in SI units are to be regarded as the standard.

1.3 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

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limitations prior to use. (Warning—Fresh hydraulic cementitious mixtures are caustic and may cause chemical burns to skin and tissue upon prolonged exposure.²)

2. Referenced Documents

2.1 ASTM Standards:

C 33 Specification for Concrete Aggregates³

C 39 Test Method for Compressive Strength of Cylindrical Concrete Specimens³

C 109/C 109M Test Method for Compressive Strength of Hydraulic-Cement Mortars (Using 2-in. or 50-mm Cube Specimens)⁴

C 114 Test Methods for Chemical Analysis of Hydraulic Cement⁴

C 115 Test Method for Fineness of Portland Cement by the Turbidimeter⁴

C 138 Test Method for Density, (Unit Weight), Yield, and Air Content (Gravimetric) of Concrete³

C 143 Test Method for Slump of Hydraulic Cement Concrete³

C 150 Specification for Portland Cement⁴

C 151 Test Method for Autoclave Expansion of Portland Cement⁴

C 175 Specifications for Air-Entraining Portland Cement⁵

C 185 Test Method for Air Content of Hydraulic Cement Mortar⁴

C 187 Test Method for Normal Consistency of Hydraulic Cement⁴

C 191 Test Method for Time of Setting of Hydraulic Cement by Vicat Needle⁴

C 192 Practice for Making and Curing Concrete Test Specimens in the Laboratory³

C 204 Test Method for Fineness of Portland Hydraulic Cement by Air Permeability Apparatus⁴

C 231 Test Method for Air Content of Freshly Mixed Concrete by the Pressure Method³

C 293 Test Method for Flexural Strength of Concrete (Using Simple Beam with Center-Point Loading)³

~~C 595/C 595M—Specification 595~~ Specification for Blended Hydraulic Cements⁴

C 596 Test Method for Drying Shrinkage of Mortar Containing Portland Hydraulic Cement⁴

C 666 Test Method for Resistance of Concrete to Rapid Freezing and Thawing³

C 1157 Performance Specification for ~~Blended~~ Hydraulic Cement⁴

2.2 ACI Standards:⁶

ACI 211.1-77 Recommended Practice for Selecting Proportions for Normal and Heavyweight Concrete

3. Materials

3.1 Cements:

3.1.1 In cases where it is desired that the proposed air-entraining addition be accepted for general use, tests shall be made on six lots of cement ground at cement plants, using commercial grinding equipment. From each of three different samples of clinkers, two lots of cement shall be ground, representing respectively: a Type I portland cement containing not less than 9 % tricalcium aluminate (C₃A), calculated as specified in Table 1 of Specification C 150 and a Type II and a Type III portland cement conforming to Specification C 150. One lot, the “control” shall be ground without the proposed air-entraining addition; the proposed addition shall be interground with the other lot, using the addition in such amounts as to produce the air/entrainment required in Specification C 150. Not more than two of the three clinkers shall be produced by or ground at the same mill.

3.1.2 In cases where it is desired that the proposed air-entraining addition be limited in use to specific types of cement in specific cement manufacturing plants, either or both less in number than required in 3.1.1, the tests and test procedure shall be as specified in 3.1.1, except that the number of cements to be tested shall be limited to those under specific consideration.

3.1.3 The two companion cements made from any one clinker shall be ground to the same fineness (within 50 cm²/g) as measured by the turbidimeter test (7.1.3) or (within 100 cm²/g) as measured by the air permeability apparatus, and the sulfur trioxide (SO₃) content expressed as a percentage of the cement weight and reported to the nearest 0.01 %, shall differ by no more than 0.24 for all types of cement. Each “control” cement shall comply with all of the requirements applicable to that type of cement, as prescribed in Specification C 150.

3.1.4 The percentage of each of the following constituents shall be determined for each lot of cement tested: silicon dioxide (SiO₂), aluminum oxide (Al₂O₃), iron oxide (Fe₂O₃), calcium oxide (CaO), magnesium oxide (MgO), SO₃, ignition loss, insoluble residue, sodium oxide (Na₂O), and potassium oxide (K₂O). There shall also be calculated the potential percentages of the following compounds: tricalcium silicate, dicalcium silicate, tricalcium aluminate, and tetracalcium aluminoferrite. Determinations for the

² See the section on Safety, Manual of Cement Testing, *Annual Book of ASTM Standards*, Vol 04.021.

³ *Annual Book of ASTM Standards*, Vol 04.042.

⁴ Discontinued, see 1949

⁴ *Annual Book of ASTM Standards*, Part 3, Vol 04.01.

⁵ Available from the American Concrete Institute, P.O. Box 19150, Detroit, MI 48219.

⁵ Discontinued, see 1949 *Annual Book of ASTM Standards*, Part 3.

⁶ Values for concrete without entrained air are based on

⁶ Available from the ACI Standard Recommended Practice for Selecting Proportions for Normal and Heavyweight American Concrete (ACI 211.1-77): Institute, P.O. Box 19150, Detroit, MI 48219.

percentage of addition shall be made on the cements containing the addition, using the method proposed therefore by the maker or seller of the addition.

3.2 Aggregates:

3.2.1 The fine and coarse aggregates used in the tests shall conform to the requirements of Specification C 33, except that the grading of the aggregates shall conform to the requirements given in Table 1.

3.2.2 The coarse aggregate shall be carefully separated on the 25.0-mm (1-in.), 19.0-mm (¾-in.), 12.5-mm (½-in.), 9.5-mm (⅜-in.), and 4.75-mm (No. 4) sieves, and then recombined, using equal quantities by weight of each of the resulting four sizes.

3.2.3 The fine and coarse aggregates used in the tests of any two companion cements (that is, a cement containing the addition and the corresponding “control” cement) shall each come from a single lot of such aggregate.

3.3 Reference Addition:

3.3.1 The reference addition used in the concrete mixture specified in Section 8, from which specimens will be made for tests for resistance to freezing and thawing as specified in 10.2.3, shall be any one of the four materials (Vinsol resin, Darex, N-TAIR, or Airalon) that have been declared acceptable by ASTM under the former Specifications C 175 – 48 T.

3.3.2 The reference addition to be used will be designated by the person or agency for whom the testing will be performed. If no reference addition is designated, the material known commercially as “Vinsol resin” shall be used. The Vinsol resin used shall be neutralized with 15 percent by mass of sodium hydroxide (NaOH). The air contents of the concrete containing the reference addition and the concrete containing the proposed addition shall agree within 0.5 percentage points.

3.3.3 The reference addition, used as an admixture, is intended for use with control cements only in concrete for freezing-and-thawing tests to establish the durability factor by means of which the concretes containing the addition under test may be evaluated.

3.3.4 Preparation of Standard Reference Solutions:

3.3.4.1 Place 50 g (total solids in the case of solution or pastes) of the designated reference addition in 500 mL of freshly distilled water in a 1000-mL flask and mix thoroughly until the solids are completely dissolved or the paste or solution is uniformly diluted. After surface foam has been dissipated, dilute to 1000 mL and mix thoroughly.

3.3.4.2 In the case of Vinsol resin, the neutralized solution shall be made as follows: Dissolve 7.50 g of cp NaOH in 100 mL of distilled water. Add a few drops of this solution to 300 to 350 mL of distilled water contained in a 600-mL beaker. Add 50.00 g of dry, unneutralized Vinsol resin in pulverized form to the beaker and stir until all of the resin is wetted and well dispersed. Then add all of the NaOH solution to this suspension and stir until all of the resin is in solution. Transfer to a measuring flask, dilute to 1000 mL and mix thoroughly. From this stock standard solution prepare a dilute standard solution by diluting 100 mL of the stock solution to 1000 mL.

4. General Requirements

4.1 Air-entraining additions shall conform to the respective requirements prescribed in this specification.

4.2 The trade name, source, and character of the material, and means for the quantitative identification of the proposed addition in the finished cement, shall be furnished by the maker or seller of the addition, and that information shall form a part of the record of tests of the addition.

4.3 Air-entraining additions shall be evaluated by testing cements ground with and without the additions. The cements ground without the additions shall be referred to in this specification as “control” cements.

4.4 An air-entraining addition under this specification, when interground with hydraulic cement, shall produce a cement that complies with the appropriate Specifications C 150, C 595, or C 1157 and when evaluated by the results of tests made according to the procedures herein described, shall also comply with the following requirements as to the effect of the addition on the properties of the cement:

4.4.1 The time of setting of cement containing the addition shall not vary from the time of setting of the respective “control” cement by more than 50 %.

4.4.2 The percentage autoclave expansion for cement containing the addition shall not exceed the percentage autoclave expansion for the corresponding “control” cement by more than 0.1.

TABLE 1 Grading Requirements of Aggregates

Sieve	Percentage Passing
Fine Aggregate	
4.75-mm (No. 4)	100
1.18-mm (No.16)	65 to 75
300-µm (No. 50)	15 to 20
150-µm (No. 100)	2 to 5
Coarse Aggregate	
25.0-mm (1-in.)	100
19.0-mm (¾-in.)	75
12.5-mm (½-in.)	50
9.5-mm (⅜-in.)	25
4.75-mm (No.4)	0

4.4.3 The compressive strength of standard mortar cubes made with cement containing the addition shall be not less than 80 % of the compressive strength of similar cubes made with the corresponding “control” cement.

4.4.4 The percentage length change of air-stored mortar bars made with cement containing the addition, based on an initial measurement at the age of 7 days, and expressed as a percentage change in length, shall be not more than 0.01 greater than that of similar mortar bars made with the corresponding “control” cement and similarly tested.

4.4.5 The percentage of air entrained in the concrete made with cement containing the addition shall exceed by at least 2.5 the percentage air in similar concrete prepared with the corresponding “control” cement. (See 3.1.1 for the limitation of air-entraining properties of the “control” cement.)

4.4.6 The compressive strength of the concrete made with cement containing the addition shall be not less than 80 % of the compressive strength of similar concrete made with the corresponding “control” cement.

4.4.7 The flexural strength of concrete made with cement containing the addition shall be not less than 85 % of the flexural strength of corresponding concrete made with the “control” cement.

4.4.8 In the freezing and thawing test, the durability factor of the concrete made with the cement containing the proposed addition shall be not less than 80 % of the durability factor of similar concrete made with the corresponding “control” cement and containing the reference addition as specified in 3.3. (See 11.1.3 for the method of calculating the durability factor.)

5. Sampling Cement

5.1 Samples of the plant-ground cements shall be obtained during grinding. Prior to the commencement of the sampling of a given lot of cement, the mill shall have run for approximately 4 h to establish equilibrium. Notes shall be kept as to the rate and continuity of the feed of the addition, the form in which the addition is used, strength of the solution, and the mill temperature. Fineness of the grinding should be checked during the grinding.

5.2 The quantity of sample shall be not less than 272 kg (600 lb) for the cement containing the proposed addition and for the corresponding control cement.

5.3 As the cement samples are secured, they shall be placed in metal drums provided with gasket-fitted lids. The drums shall be tightly closed at the end of the sampling period. Prior to use, the samples of a given lot of cement shall be well blended to form a uniform, representative composite.

6. Test Methods

6.1 Determine the properties enumerated in this specification in accordance with the methods prescribed in Sections 7-11.

7. Tests on Cement

7.1 Test the cement samples in accordance with the following methods:

7.1.1 *Chemical Analysis of Cement*—Test Methods C 114.

7.1.2 *Determination of Addition in the Finished Cement*—Determine the percentage of the addition in the finished cement by the method furnished by the manufacturer or seller of the proposed addition. The method shall be adequate for the qualitative and quantitative determination of the addition in the finished cement, and shall be fully described in the report of the tests on the addition.

7.1.3 *Fineness of Cement*—Test Method C 115 or C 204.

7.1.4 *Normal Consistency*—Test Method C 187.

7.1.5 *Time of Setting*—Determine the time of setting with the Vicat needle, in accordance with Test Method C 191.

7.1.6 *Autoclave Expansion*—Test Method C 151.

7.1.7 *Air Content of Mortar*—Test Method C 185.

7.1.8 *Compressive Strength of Mortar*—Test Method C 109/C 109M.

7.1.9 *Length Change of Mortar*—For each lot of cement, determine the length change of mortar bars according to Test Method C 596 except, after demolding, cure the specimens in saturated lime water for 6 days (7 days total age) and measure the length of each specimen at 7 days of age. Then, place the specimens in air storage for the remainder of the test period. Obtain a length comparator reading for each specimen at 28 days, 180 days, and 365 days of total age. Report the length change for each bar as a percentage of its length at 7 days of age.

8. Concrete Mixtures

8.1 *Preparation and Weighing*—Prepare all materials used in preparing the concrete mixtures, and make all weighings, as prescribed in Practice C 192. Report the amount of mixing water on the basis of saturated surface-dry aggregates.

8.2 *Proportions*—Design one concrete mixture, having an actual cement content of $307 \pm 3 \text{ kg/m}^3$ ($517 \pm 5 \text{ lb/yd}^3$), and use this mixture in all of the concrete tests specified herein. Adjust the water content of the mixture to provide concrete having a consistency equal to a $64 \pm 13\text{-mm}$ ($2\frac{1}{2} \pm \frac{1}{2}\text{-in.}$) slump. Adjust the ratio of fine aggregate to total aggregate to the optimum for

concrete to be consolidated by hand-rodding (suggested trial values⁷ for the percentage of fine aggregate in the total aggregate, by absolute volume, are shown in Table 2).

TABLE 2 Fine Aggregate in Total Aggregate, percent

Coarse Aggregate, Maximum Size 25 mm (1 in.)	Concrete Without Entrained Air	Concrete With Entrained Air
	Angular	45
Rounded	40	36

8.3 *Mixing of Concrete*—Mix the concrete in accordance with Practice C 192 except as follows: Hand mixing shall not be permitted. The rated capacity of the mixer shall be not more than twice the size of the batch used. Use a preliminary (buttering) batch of the same proportions as the test batch to coat the mixer and discard it just prior to receiving the batch of the test concrete, which shall be mixed continuously for 2 min following the addition of the mixing water. Do not scrape the mixer after buttering. At the end of the mixing period dump the concrete without scraping from the mixer into a metal wheelbarrow or pan, and then turn with a shovel before making the slump, unit weight, and air determinations, and also before being placed in the test specimen molds.

9. Tests on Plastic Concrete

9.1 Test samples of the plastic concrete in accordance with the following methods:

9.1.1 *Consistency*—Test Method C 143.

9.1.2 *Unit Weight and Air Content*—Make a determination of the unit weight, yield, and air content for each batch, in accordance with Test Method C 138, except that the air content of the concrete may be determined in accordance with Test Method C 231.

10. Test Specimens of Hardened Concrete

10.1 *Number of Specimens*—Make six test specimens from each condition of concrete to be compared for each test and age. Make test specimens representing each test and each condition of concrete from at least three separate batches. An equal number of specimens for each variable should be made on any given day. When it is impossible to make at least one specimen for each variable on a given day, complete the mixing of the entire series of specimens in as few days as possible and repeat one of the mixes each day as a standard of comparison.

10.2 *Types of Specimens*:

10.2.1 *Compressive Strength*—Compressive strength test specimens shall be 152- by 305-mm (6- by 12-in.) cylinders, and shall be made and cured as prescribed in Practice C 192.

10.2.2 *Flexural Strength*—Make and cure flexural strength test specimens as specified in Practice C 192, except that the size of the beams shall be 76 by 101 by 406 mm (3 by 4 by 16 in.).

10.2.3 *Resistance to Freezing and Thawing*—Make and cure test specimens for resistance to freezing and thawing as specified in Practice C 192, except that the size of the beams shall be 76 by 101 by 406 mm (3 by 4 by 16 in.).

11. Tests on Hardened Concrete

11.1 Test specimens of hardened concrete, as specified in Section 10, in accordance with the following methods, and at the ages herein specified.

11.1.1 *Compressive Strength*—Use Test Method C 39, and test specimens at ages of 3, 7, and 28 days, and 3 months, except that for Type III cement a test shall be made also at 1 day.

11.1.2 *Flexural Strength*—Test Method C 293. Test flexural strength specimens at the ages of 3, 7, and 28 days, 3 months, and 1 year, except that for Type III cement a test shall be made also at 1 day.

11.1.3 *Resistance to Freezing and Thawing*—Use Test Method C 666, except that the specimens shall be tested at the end of the 28-day curing period.

12. Report

12.1 The report covering the results of the evaluation of a material proposed as an air-entraining addition under this specification shall include the following information:

12.1.1 Trade name, source, and character of the material and means for the quantitative identification of the proposed addition in the finished cement—all as furnished by the manufacturer or seller of the addition.

⁷ Values for concrete without entrained air are based on the ACI Standard Recommended Practice for Selecting Proportions for Normal and Heavyweight Concrete (ACI 211.1-77).

12.1.2 Identification of the cements as to their type. General geographical location of mills where the test lots of cements were ground. (Company and brand names need not be given.)

12.1.3 Detailed results of all analyses and tests prescribed in this specification, as well as pertinent information required.

12.1.4 Comparison of test results to determine compliance with the requirements prescribed in 4.4.

12.1.5 Name and location of the laboratory or laboratories that made the tests covered by the report.

12.1.6 A statement indicating whether the evaluation was made to establish acceptability of the addition for general use (3.1.1), or whether the evaluation was made to establish acceptability for use with specific types of cement in specific cement mills (3.1.2). In the latter case, the type or types of cement and the brand names and location of mills that are covered by the tests shall be given.

13. Keywords

13.1 addition; air entraining; hydraulic cement

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