



## Standard Test Method for Air-Entraining Admixtures for Concrete<sup>1</sup>

This standard is issued under the fixed designation C 233; 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 ( $\epsilon$ ) 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 This test method covers the testing of materials proposed for use as air-entraining admixtures in the field.

1.2 The values stated in SI units are to be regarded as the standard. The values given in parentheses are for information purposes only.

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 limitations prior to use.*

1.4 The text of this test method references notes and footnotes which provide explanatory material. These notes and footnotes (excluding those in tables and figures) shall not be considered as requirements of the standard.

### 2. Referenced Documents

#### 2.1 ASTM Standards:

- C 33 Specification for Concrete Aggregates<sup>2</sup>
- C 39 Test Method for Compressive Strength of Cylindrical Concrete Specimens<sup>2</sup>
- C 78 Test Method for Flexural Strength of Concrete (Using Simple Beam with Third-Point Loading)<sup>2</sup>
- C 136 Test Method for Sieve Analysis of Fine and Coarse Aggregates<sup>2</sup>
- C 143/C 143M Test Method for Slump of Hydraulic Cement Concrete<sup>2</sup>
- C 150 Specification for Portland Cement<sup>3</sup>
- C 157 Test Method for Length Change of Hardened Hydraulic-Cement Mortar and Concrete<sup>2</sup>
- C 172 Practice for Sampling Freshly Mixed Concrete<sup>2</sup>
- C 173 Test Method for Air Content of Freshly Mixed Concrete by the Volumetric Method<sup>2</sup>
- C 185 Test Method for Air Content of Hydraulic Cement Mortar<sup>3</sup>
- C 192/C 192M Test Method for Making and Curing Concrete Test Specimens in the Laboratory<sup>2</sup>

C 231 Test Method for Air Content of Freshly Mixed Concrete by the Pressure Method<sup>2</sup>

C 232 Test Methods for Bleeding of Concrete<sup>2</sup>

C 260 Specification for Air-Entraining Admixtures for Concrete<sup>2</sup>

C 403/C 403M Test Method for Time of Setting of Concrete Mixtures by Penetration Resistance<sup>2</sup>

C 666 Test Method for Resistance of Concrete to Rapid Freezing and Thawing<sup>2</sup>

C 670 Practice for Preparing Precision and Bias Statements for Test Methods for Construction Materials<sup>2</sup>

D 75 Practice for Sampling Aggregates<sup>4</sup>

D 1193 Specification for Reagent Water<sup>5</sup>

E 70 Test Method for pH of Aqueous Solutions with the Glass Electrode<sup>6</sup>

#### 2.2 ACI Standards:

ACI 211.1 Recommended Practice for Selecting Proportions for Normal, Heavyweight, and Mass Concrete<sup>7</sup>

### 3. Significance and Use

3.1 This test method is used to develop data for comparison with the requirements of Specification C 260. These tests are based on arbitrary stipulations permitting highly standardized testing in the laboratory, and are not intended to simulate actual job conditions.

### 4. Materials

4.1 *Cement*—The cement used in any series of tests shall be either the cement proposed for specific work in accordance with 4.4, a Type I or Type II cement conforming to Specification C 150, or a blend of two or more cements, in equal parts. Each cement of the blend shall conform to the requirements of either Type I or Type II, Specification C 150. If a blend of cements is used, it shall be a combination which produces an air content of less than 10 % when tested in accordance with Test Method C 185 (Note 3).

4.2 *Aggregates*—Except when tests are made in accordance with 4.4, using the aggregates proposed for specific work, the fine and coarse aggregates used in any series of tests shall come

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<sup>2</sup> *Annual Book of ASTM Standards*, Vol 04.02.

<sup>3</sup> *Annual Book of ASTM Standards*, Vol 04.01.

<sup>4</sup> *Annual Book of ASTM Standards*, Vol 04.03.

<sup>5</sup> *Annual Book of ASTM Standards*, Vol 11.01.

<sup>6</sup> *Annual Book of ASTM Standards*, Vol 15.05.

<sup>7</sup> American Concrete Institute Manual of Concrete Practice, Part 1, pp. 211-1 to 211-38 (1993).

from single lots of well-graded, sound materials that conform to the requirements of Specification C 33, except that the grading of the aggregates shall conform to the following requirements:

4.2.1 *Fine Aggregate Grading*—The fine aggregate shall meet the requirements for the fine aggregate in Specification C 33.

4.2.2 *Coarse Aggregate Grading*—The coarse aggregate shall meet the requirements for size number 57 of Specification C 33.

NOTE 1—Take care in loading and delivery to avoid segregation.

4.2.3 The coarse aggregate used for each set of reference concrete and comparable test admixture-treated concrete shall be essentially the same. Therefore, a set of test concrete consists of one reference concrete and as many test admixture-containing concretes as are intended to be compared to that one reference. Thus, coarse aggregate for one set shall consist of enough material for one reference concrete, the test admixture-containing concrete to be compared with that reference, and the sample for grading analysis testing.

4.2.3.1 Prepare coarse aggregate for a set, comprising a sample large enough for concrete trials, as follows: Fill tared containers, one each for a sample, a batch of reference concrete and one or more test concretes to the required mass from the aggregate stockpile. Accomplish this by starting with a scoopful into the first container and repeat this procedure until all containers have their required mass. Repeat the process for each of the three or more sets needed. One or more spare sets may be needed. See the Appendix of Practice D 75, Sampling from Stockpiles, and the Manual of Aggregate and Concrete Testing<sup>2</sup> for guidance for conditions and procedures.

4.2.4 Test coarse aggregate samples representing each set by Test Method C 136 requirements for the sieves shown below. Discard any set for which the sample does not comply with Size 57. Average test results for samples which comply with Size 57 for each sieve size. Discard any set for which the sample deviates from this average by more than the amount shown in column 3. Continue the process of preparation, testing and averaging until sufficient sets of aggregate within tolerance are obtained.

Sieve	Specification C 33, No. 57 Percent Passing	Maximum variation from average/passing
37.5-mm	100	00
25.0-mm	95 to 100	1.0
12.5-mm	25 to 60	4.0
4.75-mm	0 to 10	4.0
2.36-mm	0 to 5	1.0

NOTE 2—All of the results required for demonstrating compliance under this specification are dependent on the uniformity of the aggregate samples prepared and used. Careful, skilled and well-supervised work is essential.

4.3 *Reference Admixture*—For this test method, unless otherwise requested by the purchaser, the reference admixture used in the concrete mixture specified in Section 4 shall be

“neutralized Vinsol resin.”<sup>8</sup>

4.4 *Materials for Tests for Specific Uses*—When it is desired to test an air-entraining admixture for use in specific work, the cement and aggregates used shall be representative of those proposed for use in the work, and the concrete mixtures shall be designed to have the cement content specified for use in the work (Note 3). If the maximum size of coarse aggregate is greater than 25.0 mm (1 in.), the freshly mixed concrete shall be screened over a 25.0-mm (1-in.) sieve prior to fabricating the test specimens in accordance with the wet sieving procedure described in Practice C 172.

4.5 *Preparation and Weighing*—All materials shall be prepared and all weighings shall be made as prescribed in Test Method C 192/C 192M.

NOTE 3—It is recommended that whenever practicable, tests be made in accordance with 4.4 using the cement and pozzolanic or chemical admixtures, if any, proposed for specific work.

## 5. Concrete Mixtures

5.1 *Proportions*—Using ACI 211.1, all concrete shall be proportioned to conform to the following requirements:

5.1.1 The cement content shall be  $307 \pm 3 \text{ kg/m}^3$  ( $517 \pm 5 \text{ lb/yd}^3$ ) except when tests are being made for specific uses (see 4.4).

5.1.2 The first trial mixture shall contain the amount of coarse aggregate shown in Table 6.3.6 of ACI Recommended Practice 211.1 for the maximum size of aggregate and for the fineness modulus of the sand being used.

NOTE 4—Values in Table 6.3.6 of ACI Recommended Practice 211.1-77 are intended to ensure workable mixtures with the least favorable combinations of aggregate likely to be used. It is suggested, therefore, that for a closer approximation of the proportions required for this test, the values selected from Table 6.3.6 be increased by about 7 % for the first trial mixture.

5.1.3 The air content used in the computation of proportions for all concrete shall be 5.5 % except where the admixture under test is for use in specific work (see 4.4). In this case the air content used in selecting proportions shall be the median of the range to be permitted in the work. If lightweight aggregates are to be used in specific work, the unit weight of concrete used in selecting proportions shall be the median of the range permitted in the work.

5.1.4 The water content and sand content shall be adjusted to obtain a slump of  $90 \pm 15 \text{ mm}$  ( $3\frac{1}{2} \pm \frac{1}{2} \text{ in.}$ ). The workability of the concrete mixture shall be suitable for consolidation by hand rodding and the concrete mixture shall have the minimum water content possible. These conditions shall be achieved by final adjustments in the proportion of fine aggregate to total aggregate, in the amount of total aggregate, or both, while maintaining the yield and slump in the required ranges.

5.2 *Conditions*—Concrete mixtures shall be prepared both with the air-entraining admixture under test and with the

<sup>8</sup> Vinsol resin is manufactured by Hercules Inc., Wilmington, DE. Neutralization may be accomplished by treating 100 parts of the Vinsol resin with 9 to 15 parts of NaOH by weight. In an aqueous solution, the ratio of water to the resin shall not exceed 12:1 by weight.

reference admixture. The admixtures shall be added in the amounts necessary to produce the air content selected in accordance with 5.1.3 within a tolerance of  $\pm 0.5\%$  of the volume of concrete.

## 6. Mixing

6.1 Machine mix the concrete as prescribed in Test Method C 192/C 192M.

## 7. Tests and Properties of Freshly Mixed Concrete

7.1 Test samples of freshly mixed concrete from at least three separate batches for each condition of concrete in accordance with the following methods and the minimum number of tests shall be as prescribed in Table 1.

7.1.1 *Slump*—Test Method C 143/C 143M.

7.1.2 *Air Content*—Test Method C 231. When lightweight aggregates are used under the provisions of 4.4, use Test Method C 173.

7.1.3 *Bleeding*—Test Methods C 232.

7.1.4 *Time of Setting*—Test Method C 403/C 403M, except that the temperature of each of the ingredients of the concrete mixtures, just prior to mixing, and the temperature at which the time of setting specimens are stored during the test period shall be  $23.0 \pm 2^\circ\text{C}$  ( $73 \pm 3^\circ\text{F}$ ).

## 8. Preparation of Test Specimens

8.1 Specimens for test of hardened concrete, representing each test and age of test and each condition of concrete being compared, shall be made from at least three separate batches, and the minimum number of specimens shall be as prescribed in Table 1. On a given day at least one specimen shall be made for each test and age of test from each condition of concrete except that at least two specimens for the freezing and thawing test shall be made from each condition of concrete. The preparation of all specimens shall be completed in three days of mixing.

8.2 *Manifestly Faulty Specimens*—Each group of specimens representing a given test or a given age of test, including tests of freshly mixed concrete, shall be examined visually before or during the test, or both, whichever is appropriate. Discard any specimen found to be manifestly faulty by such examination without testing. Visually examine all specimens representing a given test at a given age after testing, and should any specimen

be found to be manifestly faulty, the test results thereof shall be disregarded. Should more than one specimen representing a given test at a given age be found manifestly faulty, either before or after testing, the entire test shall be disregarded and repeated. The test result reported shall be the average of the individual test results of the specimens tested or, in the event that one specimen or one result has been discarded, it shall be the average of the test results of the remaining specimens.

## 9. Test Specimens of Hardened Concrete

9.1 *Number of Specimens*—Make six or more test specimens for the freezing and thawing test and three or more test specimens for each other type of test and age of test specified in Table 1 for each condition of concrete to be compared.

9.2 *Types of Specimens*—Prepare specimens made from concrete with and without the air-entraining admixture under test in accordance with the following:

9.2.1 *Compressive Strength*—Make and cure test specimens in accordance with Test Method C 192/C 192M.

9.2.2 *Flexural Strength*—Make and cure test specimens in accordance with Test Method C 192/C 192M.

9.2.3 *Resistance to Freezing and Thawing*—Test specimens shall consist of prisms made and cured in accordance with the applicable requirement of Test Method C 192/C 192M. Test specimen dimensions shall be as required by Test Method C 666. Make one set of specimens from the concrete mixture containing the air-entraining admixture under test and from the reference concrete mixture, the air content of each mixture being as specified in 5.2.

9.2.4 *Length Change*—Make and cure test specimens in accordance with Test Method C 157. The moist-curing period, including the period in the molds, shall be 14 days.

## 10. Tests on Hardened Concrete

10.1 Test specimens of hardened concrete in accordance with the following test methods:

10.1.1 *Compressive Strength*—Test Method C 39. Test specimens at ages of 3, 7, and 28 days. Calculate the compressive strength of the concrete containing the admixture under test as a percentage of the compressive strength of the reference concrete as follows:

10.1.1.1 Divide the average compressive strength of the specimens made from the concrete containing the admixture

**TABLE 1 Types and Minimum Number of Specimens and Tests**

Test	Number of Types of Specimens <sup>A</sup>	Number of Test Ages	Number of Conditions of Concrete <sup>B</sup>	Minimum Number of Specimens
Slump	1	1	2	C
Air content	1	1	2	C
Bleeding	1	1	2	6
Time of setting	1	D	2	6
Compressive strength	1	5	2	30
Flexural strength <sup>E</sup>	1	5	2	30
Freezing and thawing	1	1	2	12 <sup>F</sup>
Length change <sup>E</sup>	1	1	2	6

<sup>A</sup> See Section 7 and 9.2.

<sup>B</sup> See 4.2.

<sup>C</sup> Determined on each batch of concrete mixed.

<sup>D</sup> See 7.1.4.

<sup>E</sup> Optional tests, see 10.1.5.

<sup>F</sup> Specimens for duplicate tests from each batch.

under test at a given age of test by the average compressive strength of the specimens made from the reference concrete at the same age of test and multiply the quotient by 100.

10.1.2 *Flexural Strength*—Test Method C 78. Test specimens at ages 3, 7, and 28 days. Calculate the flexural strength of the concrete containing the admixture under test as a percentage of the flexural strength of the reference concrete as follows:

10.1.2.1 Divide the average flexural strength of the specimens made from the concrete containing the admixture under test at a given age of test by the average flexural strength of the specimens made from the reference concrete at the same age of test, and multiply the quotient by 100.

10.1.3 *Resistance to Freezing and Thawing*—Procedure A of Test Method C 666. Place specimens under test at the age of 14 days.

10.1.4 *Length Change*—Test Method C 157. The drying period shall be 14 days.

10.1.5 The flexural strength and length change tests are applicable only when specifically required by the purchaser.

## 11. Check Tests for Uniformity

11.1 The check tests enumerated in Specification C 260 in the section on Optional Uniformity Requirements shall be determined as follows:

11.1.1 *pH*—The pH of liquid air-entraining admixtures shall be determined in accordance with Test Method E 70. Non-liquid admixtures shall be prepared in solution to determine pH. Unless there is reason to do otherwise, dissolve the material in water in the proportions specified for job use as shown on the package or in other manufacturer's instructions. The temperature of the check test sample shall be within  $\pm 1^\circ\text{C}$  ( $\pm 2^\circ\text{F}$ ) of that for the acceptance sample and preferably in the range of 21 to  $27^\circ\text{C}$  (70 to  $80^\circ\text{F}$ ).

11.1.2 *Air Content of Mortar*—Using the same amounts of successive lots of air-entraining admixtures with the same cement, determine the air contents of mortars in accordance with Test Method C 185. The air-entraining admixture shall be combined with the mixing water prior to the start of the mixing procedure. The determinations for both the check test sample and acceptance sample shall be made on the same day.

## 12. Procedure for Residue by Oven Drying

12.1 Determine the mass of an aluminum dish (about 57 mm diameter, 15 mm height, and about 1 g in weight) to the nearest 0.0001 g. Using a pipet, evenly distribute 1 ml of the liquid air entraining admixture in the dish, and weigh to the nearest 0.0001 g. Place the weighing dish in a drying oven (12.2). Dry for  $25 \pm 2$  min at  $125 \pm 1^\circ\text{C}$ . At the end of the drying period transfer the weighing dish to a desiccator, cool to room temperature, and weigh to the nearest 0.0001 g.

12.2 The drying oven shall be either a forced circulation type or one with provision for free access of air. There shall be precise control of temperature and time of drying so that the degree of volatilization of the material other than water from sample to sample will not vary.

### 12.3 Calculation:

12.3.1 Record the following weights:

$M_1$  = mass of weighing dish and admixture prior to heating,

$M_2$  = mass of empty weighing dish,

$M_3 = M_1 - M_2$  = mass of sample,

$M_4$  = mass of weighing dish and dried residue, and

$M_5 = M_4 - M_2$  = mass of dried residue.

12.3.2 Calculate the residue by using the following equation:

$$\text{Residue by oven drying (\% by mass)} = (M_5 \times 100)/M_3 \quad (1)$$

## 13. Report

13.1 Report the following information:

13.1.1 Results of the tests specified in this method as compared with the requirements of Specification C 260,

13.1.2 Brand name, manufacturer's name and lot number, character of the material, and quantity represented by the sample of the admixture under test,

13.1.3 Brand name, manufacturer's name, and other data on the reference admixture,

13.1.4 Brand name, manufacturer's name, type, and test data on the portland cement or cements used,

13.1.5 Description of, and test data on the fine and coarse aggregates used,

13.1.6 Detailed data on the concrete mixtures used, including amounts and proportions of admixtures used, actual cement factors, water-cement ratios, ratios of fine to total aggregate, consistency, and air content.

13.1.7 In reporting on check tests for uniformity, report both the initial and current air contents of mortar for the acceptance sample, and the air content of the check test sample, all as determined by Test Method C 185.

## 14. Precision and Bias<sup>9</sup>

14.1 *Precision:*

14.1.1 The single-laboratory coefficient of variation of residue by oven drying has been found to be 0.79 %. Therefore, the results of two properly conducted tests on the same material in the same laboratory are not expected to differ by more than 2.24 % of their average.<sup>10</sup>

NOTE 5—As an example, two tests conducted on the same material yield residues by oven drying of 6.14 % and 6.04 %, respectively. The average of these two measurements is 6.09 %. The acceptable range of results is then 2.24 % of 6.09 % or  $\pm 0.136$  %. As the difference between 6.14 % and 6.04 % is 0.10 % the results are within the acceptable range.

14.1.2 The multilaboratory coefficient of variation of residue by oven drying has been found to be 2.35 %. Therefore, the results of two properly conducted tests on the same material in different laboratories are not expected to differ by more than 6.65 % of their average.<sup>10</sup>

14.1.3 Other procedures referenced in this test method use results obtained from other ASTM test methods listed in Section 2. These documents are to be referred to for their respective precision statements.

<sup>9</sup> A copy of the research report used to develop the precision statement is available from ASTM headquarters. Request RR: C09-1005.

<sup>10</sup> These numbers represent, respectively, the (1s %) and (d2s %) limits as described in Practice C 670.

14.2 *Bias*—Since there is no accepted reference material suitable for determining the bias of this test method, no statement on bias is made.

## 15. Keywords

15.1 air content; air-entraining admixture; cement; concrete; pH; residue; specific gravity

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