



# Standard Specification for Chemical Admixtures for Concrete<sup>1</sup>

This standard is issued under the fixed designation C 494/C 494M; 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.*

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<sup>ε1</sup> NOTE—Range value in 12.2.2 was editorially corrected July 2001.

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## 1. Scope

1.1 This specification covers materials for use as chemical admixtures to be added to hydraulic-cement concrete mixtures in the field for the purpose or purposes indicated for the seven types as follows:

- 1.1.1 *Type A*—Water-reducing admixtures,
- 1.1.2 *Type B*—Retarding admixtures,
- 1.1.3 *Type C*—Accelerating admixtures,
- 1.1.4 *Type D*—Water-reducing and retarding admixtures,
- 1.1.5 *Type E*—Water-reducing and accelerating admixtures,
- 1.1.6 *Type F*—Water-reducing, high range admixtures, and
- 1.1.7 *Type G*—Water-reducing, high range, and retarding admixtures.

1.2 This specification stipulates tests of an admixture with suitable concreting materials as described in 11.1-11.3 or with cement, pozzolan, aggregates, and an air-entraining admixture proposed for specific work (11.4). Unless specified otherwise by the purchaser, the tests shall be made using concreting materials as described in 11.1-11.3.

NOTE 1—It is recommended that, whenever practicable, tests be made using the cement, pozzolan, aggregates, air-entraining admixture, and the mixture proportions, batching sequence, and other physical conditions proposed for the specific work (11.4) because the specific effects produced by chemical admixtures may vary with the properties and proportions of the other ingredients of the concrete. For instance, Types F and G admixtures may exhibit much higher water reduction in concrete mixtures having higher cement factors than that listed in 12.1.1.

Mixtures having a high range water reduction generally display a higher rate of slump loss. When high-range admixtures are used to impart increased workability (6 to 8-in. slump [150 to 200-mm]), the effect may be of limited duration, reverting to the original slump in 30 to 60 min depending on factors normally affecting rate of slump loss. The use of chemical admixtures to produce high-slump (flowing) concrete is covered by Specification C 1017.

NOTE 2—The purchaser should ensure that the admixture supplied for use in the work is equivalent in composition to the admixture subjected to test under this specification (see Section 6, Uniformity and Equivalence).

NOTE 3—Admixtures that contain relatively large amounts of chloride may accelerate corrosion of prestressing steel. Compliance with the

requirements of this specification does not constitute assurance of acceptability of the admixture for use in prestressed concrete.

1.3 This specification provides for three levels of testing.

1.3.1 *Level 1*—During the initial approval stage, proof of compliance with the performance requirements defined in Table 1 demonstrates that the admixture meets the requirements of this specification. Uniformity and equivalence tests of Section 6 shall be carried out to provide results against which later comparisons can be made.

1.3.2 *Level 2*—Limited retesting is described in 5.2, 5.2.1 and 5.2.2. Proof of compliance with the requirements of Table 1 demonstrates continued conformity of the admixture with the requirements of the specification.

1.3.3 *Level 3*—For acceptance of a lot or for measuring uniformity within or between lots, when specified by the purchaser, the uniformity and equivalence tests of Section 6 shall be used.

1.4 The values stated in either inch-pound or SI units shall be regarded separately as standard. The values stated in each system may not be exact equivalents; therefore, each system must be used independently of the other, without combining values in any way.

1.5 The text of this standard 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.

1.6 The following precautionary caveat pertains only to the test method sections, Sections 11-18 of this Specification: *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.*

## 2. Referenced Documents

### 2.1 ASTM Standards:

C 33 Specification for Concrete Aggregates<sup>2</sup>

C 39/C 39M Test Method for Compressive Strength of Cylindrical Concrete Specimens<sup>2</sup>

C 78 Test Method for Flexural Strength of Concrete (Using

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<sup>1</sup> This specification is under the jurisdiction of ASTM Committee C09 on Concrete Aggregates and is the direct responsibility of Subcommittee C9.23 on Chemical Admixtures.

Current edition approved Oct. 10, 1999. Published December 1999. Originally published as C 494 – 62. Last previous edition C 494 – 99.

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

**TABLE 1 Physical Requirements<sup>A</sup>**

	Type A, Water Reducing	Type B, Retarding	Type C, Accelerating	Type D, Water Reducing and Retarding	Type E, Water Reducing and Accelerating	Type F, Water Reducing, High Range	Type G, Water Reducing, High Range and Retarding
Water content, max, % of control	95	...	...	95	95	88	88
Time of setting, allowable deviation from control, h:min:							
Initial: at least	...	1:00 later	1:00 earlier	1:00 later	1:00 earlier	...	1:00 later
not more than	1:00 earlier nor 1:30 later	3:30 later	3:30 earlier	3:30 later	3:30 earlier	1:00 earlier nor 1:30 later	3:30 later
Final: at least	...	...	1:00 earlier	...	1:00 earlier	...	...
not more than	1:00 earlier nor 1:30 later	3:30 later	...	3:30 later	...	1:00 earlier nor 1:30 later	3:30 later
Compressive strength, min, % of control: <sup>B</sup>							
1 day	...	...	...	...	...	140	125
3 days	110	90	125	110	125	125	125
7 days	110	90	100	110	110	115	115
28 days	110	90	100	110	110	110	110
6 months	100	90	90	100	100	100	100
1 year	100	90	90	100	100	100	100
Flexural strength, min, % control: <sup>B</sup>							
3 days	100	90	110	100	110	110	110
7 days	100	90	100	100	100	100	100
28 days	100	90	90	100	100	100	100
Length change, max shrinkage (alternative requirements): <sup>C</sup>							
Percent of control	135	135	135	135	135	135	135
Increase over control	0.010	0.010	0.010	0.010	0.010	0.010	0.010
Relative durability factor, min <sup>D</sup>	80	80	80	80	80	80	80

<sup>A</sup> The values in the table include allowance for normal variation in test results. The object of the 90 % compressive strength requirement for a Type-B admixture is to require a level of performance comparable to that of the reference concrete.

<sup>B</sup> The compressive and flexural strength of the concrete containing the admixture under test at any test age shall be not less than 90 % of that attained at any previous test age. The objective of this limit is to require that the compressive or flexural strength of the concrete containing the admixture under test shall not decrease with age.

<sup>C</sup> Alternative requirements, see 17.1.4, % of control limit applies when length change of control is 0.030 % or greater; increase over control limit applies when length change of control is less than 0.030 %.

<sup>D</sup> This requirement is applicable only when the admixture is to be used in air-entrained concrete which may be exposed to freezing and thawing while wet.

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 138 Test Method for Unit Weight, Yield, and Air Content (Gravimetric) of Concrete<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/C 157M Test Method for Length Change of Hardened Hydraulic-Cement Mortar and Concrete<sup>2</sup>  
 C 183 Practice for Sampling and the Amount of Testing of Hydraulic Cement<sup>3</sup>  
 C 192/C 192M Practice 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 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 1017 Specification for Chemical Admixtures for Use in Producing Flowing Concrete<sup>2</sup>  
 D 75 Practice for Sampling Aggregates<sup>2</sup>  
 D 1193 Specification for Reagent Water<sup>4</sup>  
 E 100 Specification for ASTM Hydrometers<sup>5</sup>  
 Manual of Aggregate and Concrete Testing<sup>2</sup>  
 2.2 *American Concrete Institute Standard:*  
 ACI 211.1–91 Standard Practice for Selecting Proportions for Normal, Heavyweight, and Mass Concrete<sup>6</sup>

### 3. Terminology

#### 3.1 Definitions:

3.1.1 *accelerating admixture*—an admixture that accelerates the setting and early strength development of concrete.

3.1.2 *retarding admixture*—an admixture that retards the setting of concrete.

<sup>4</sup> *Annual Book of ASTM Standards*, Vol 11.01.

<sup>5</sup> *Annual Book of ASTM Standards*, Vol 14.03.

<sup>6</sup> Available from the American Concrete Institute, 38800 Country Club Drive, Farmington Hills, MI 48331.

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

3.1.3 *water-reducing admixture*—an admixture that reduces the quantity of mixing water required to produce concrete of a given consistency.

3.1.4 *water-reducing admixture, high range*—an admixture that reduces the quantity of mixing water required to produce concrete of a given consistency by 12 % or greater.

3.1.5 *water-reducing and accelerating admixture*—an admixture that reduces the quantity of mixing water required to produce concrete of a given consistency and accelerates the setting and early strength development of concrete.

3.1.6 *water-reducing and retarding admixture*—an admixture that reduces the quantity of mixing water required to produce concrete of a given consistency and retards the setting of concrete.

3.1.7 *water-reducing, high range, and retarding admixture*—an admixture that reduces the quantity of mixing water required to produce concrete of a given consistency by 12 % or greater and retards the setting of concrete.

#### 4. Ordering Information

4.1 The purchaser shall specify the type of chemical admixture desired.

#### 5. General Requirements

5.1 For initial compliance with this specification, test concrete in which each type of admixture shown in 1.1 is used shall conform to the respective requirements prescribed in Table 1.

5.2 The purchaser is allowed to require a limited retesting to confirm current compliance of the admixture to specification requirements. The limited retesting will cover physical properties and performance of the admixture.

5.2.1 The physical properties retesting shall consist of uniformity and equivalence tests for infrared analysis, residue by oven drying and specific gravity.

5.2.2 The performance property retesting shall consist of water content of fresh concrete, setting time and compressive strength at 3, 7 and 28 days. Purchasers having special requirements are allowed to require additional tests currently in this standard.

5.3 At the request of the purchaser, the manufacturer shall state in writing that the admixture supplied for use in the work is identical in all essential respects, including concentration, to the admixture tested under this specification.

5.4 At the request of the purchaser, when the admixture is to be used in prestressed concrete, the manufacturer shall state in writing the chloride content of the admixture and whether or not chloride has been added during its manufacture.

5.5 Tests for uniformity and equivalence, as indicated in Section 6, shall be made on the initial sample and the results retained for reference and comparison with the results of tests of samples taken from elsewhere within the lot or subsequent lots of admixture supplied for use in the work.

#### 6. Uniformity and Equivalence

6.1 When specified by the purchaser, the uniformity of a lot, or the equivalence of different lots from the same source shall be established by the use of the following requirements:

6.1.1 *Infrared Analysis*—The absorption spectra of the ini-

tial sample and the test sample, obtained as specified in 18.1, shall be essentially similar.

6.1.2 *Residue by Oven Drying (Liquid Admixtures)*—When dried as specified in 18.2, the oven-dried residues of the initial sample and of subsequent samples shall be within a range of variation of not greater than 5 percentage points.

6.1.3 *Residue by Oven Drying (Nonliquid Admixtures)*—When dried as specified in 18.3, the oven-dried residues of the initial sample and of the subsequent samples shall be within a range of variation not greater than 4 percentage points.

6.1.4 *Specific Gravity (Liquid Admixtures)*—When tested as specified in 18.4, the specific gravity of subsequent test samples shall not differ from the specific gravity of the initial sample by more than 10 % of the difference between the specific gravity of the initial sample and that of reagent water at the same temperature. Reagent water conforming to Specification D 1193, Types III or IV, and prepared by distillation ion exchange, reverse osmosis, electrodialysis, or a combination of these procedures is adequate.

6.2 When the nature of the admixture or the analytical capability of the purchaser make some or all of these procedures unsuitable, other requirements for uniformity and equivalence from lot to lot or within a lot shall be established by agreement between the purchaser and the manufacturer.

#### 7. Packaging and Marking

7.1 When the admixture is delivered in packages or containers, the proprietary name of the admixture, the type under this specification, and the net weight or volume shall be plainly marked thereon. Similar information shall be provided in the shipping advices accompanying packaged or bulk shipments of admixtures.

#### 8. Storage

8.1 The admixture shall be stored in such a manner as to permit easy access for proper inspection and identification of each shipment, and in a suitable weathertight building that will protect the admixture from dampness and freezing.

#### 9. Sampling and Inspection

9.1 Every facility shall be provided the purchaser for careful sampling and inspection, either at the point of manufacture or at the site of the work, as specified by the purchaser.

9.2 Samples shall be either “grab” or “composite” samples, as specified or required by this specification. A grab sample is one obtained in a single operation. A composite sample is one obtained by combining three or more grab samples.

9.3 For the purposes of this specification, it is recognized that samples will be taken for two reasons:

9.3.1 *Quality Tests*—A sample taken for the purpose of evaluating the quality of a source or lot of admixture will be required to meet all the applicable requirements of this specification. Samples used to determine conformance with the requirements of this specification shall be composites of grab samples taken from sufficient locations to ensure that the composite sample will be representative of the lot.

9.3.2 *Uniformity and Equivalence Tests*—When specified by the purchaser, a sample taken for the purpose of evaluating the uniformity of a single lot, or equivalence of different lots

from one source shall be tested as provided in Section 6. Such samples shall be composite samples from individual lots when different lots from the same source are being compared. When the uniformity of a single lot is being determined, grab samples shall be used.

9.4 *Liquid Admixtures*—Liquid admixtures shall be agitated thoroughly immediately prior to sampling. Grab samples taken for quality or uniformity tests shall represent a unit shipment or a single production lot. Each grab sample shall have a volume of at least 1 pt [0.5 L]. A minimum of three grab samples shall be taken. Composite samples shall be prepared by thoroughly mixing the grab samples selected and the resultant mixture sampled to provide at least 1 gal [4 L] for quality tests. Grab samples shall be taken from different locations well distributed throughout the quantity to be represented.

9.4.1 Admixtures in bulk storage tanks shall be sampled equally from the upper, intermediate, and lower levels by means of drain cocks in the sides of the tanks or a weighted sampling bottle fitted with a stopper that can be removed after the bottle is lowered to the desired depth.

9.4.2 Samples shall be packaged in impermeable, airtight containers which are resistant to attack by the admixture.

9.5 *Nonliquid Admixtures*—Grab samples taken for quality or uniformity tests shall represent not more than 2 tons [2 Mg] of admixture and shall weigh at least 2 lb [have a mass of at least 1 kg]. A minimum of four grab samples shall be taken. Composite samples shall be prepared by thoroughly mixing the grab samples selected and the resultant mixture sampled to provide at least 5 lb [2.5 kg] for the composite sample. Grab samples shall be taken from different locations well distributed throughout the quantity to be represented.

9.5.1 Samples of packaged admixtures shall be obtained by means of a tube sampler as described in Practice C 183.

9.5.2 Samples shall be packaged in moisture-proof, airtight containers.

9.6 Samples shall be thoroughly mixed before testing to ensure uniformity. When recommended by the manufacturer, the entire sample of a nonliquid admixture shall be dissolved in water prior to testing.

**10. Rejection**

10.1 For initial compliance testing, the purchaser is allowed to reject the admixture if it fails to meet any of the applicable requirements for this specification.

10.2 For limited retesting, the purchaser is allowed to reject the admixture if it fails to meet any of the requirements of the Uniformity and Equivalence Section and of the applicable parts of Table 1.

10.3 An admixture stored at the point of manufacture, for more than 6 months prior to shipment, or an admixture in local storage in the hands of a vendor for more than 6 months, after completion of tests, shall be retested before use when requested by the purchaser and is allowed to be rejected if it fails to conform to any of the applicable requirements of this specification.

10.4 Packages or containers varying more than 5 % from the specified weight or volume are allowed to be rejected. If the average weight or volume of 50 packages taken at random is

less than that specified, the entire shipment is allowed to be rejected.

10.5 When the admixture is to be used in non-air-entrained concrete, it shall be rejected when the purchaser desires if the test concrete containing it has an air content greater than 3.5 %; when the admixture is to be used in air-entrained concrete, it can be rejected if the test concrete containing it has an air content greater than 7.0 %.

**TEST METHODS**

NOTE 4—These tests are based on arbitrary stipulations which make possible highly standardized testing in the laboratory and are not intended to simulate actual job conditions.

**11. Materials**

**TESTS NOT FOR A SPECIFIC USE**

11.1 *Cement*—The cement used in any series of tests shall be either the cement proposed for a specific use in accordance with 11.4, 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 when using a cement other than that proposed for specific work, the air content of the concrete made without admixture, tested as prescribed in 14.3, is more than 3.5 %, select a different cement, or blend, so that the air content of the concrete will be 3.5 % or less.

11.2 *Aggregates*—Except when tests are made in accordance with 11.4 using the aggregates proposed for a specific use, the fine and coarse aggregates used in any series of tests shall come 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:

11.2.1 *Fine Aggregate Grading:*

Sieve	Weight Percent
No. 4 [4.75-mm]	Passing 100
No. 16 [1.18-mm]	65 to 75
No. 50 [300 μm]	12 to 20
No. 100 [150 μm]	2 to 5

11.2.2 *Coarse Aggregate Grading*— The coarse aggregate shall meet the requirements for size number 57 of Specification C 33. Take care in loading and delivery to avoid segregation.

11.2.3 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.

11.2.3.1 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 for guidance for conditions and procedures.

11.2.4 Test coarse aggregate samples representing each set by 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
1 1/2 in. [37.5-mm]	100	0.0
1.00 in. [25.0-mm]	95 to 100	1.0
1/2 in. [12.5-mm]	25 to 60	4.0
No. 4 [4.75-mm]	0 to 10	4.0
No. 8 [2.36-mm]	0 to 5	1.0

NOTE 5—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.

11.3 *Air-Entraining Admixture*—Except when tests are made in accordance with 11.4 using the air-entraining admixture proposed for specific work, the air-entraining admixture used in the concrete mixtures specified in Section 12 shall be a material such that when used to entrain the specified amount of air in the concrete mixture will give concrete of satisfactory resistance to freezing and thawing. The material to be so used will be designated by the person or agency for whom the testing is to be performed. If no material is designated, “neutralized Vinsol resin”<sup>7</sup> shall be used. Accomplish neutralization by treating 100 parts of Vinsol resin with 9 to 15 parts of NaOH by mass. In an aqueous solution, the ratio of water to the resin shall not exceed 12 to 1 by mass.

**TESTS FOR SPECIFIC USES**

11.4 *Materials for Tests*—The effects of a chemical admixture on the time of setting and water requirement of concrete are known to vary with the time of its addition during the batching and mixing sequence. To test a chemical admixture for use in specific work, the cement, pozzolan, aggregates, and air-entraining admixture used shall be representative of those proposed for use in the work. Add the chemical admixture in the same manner and at the same time during the batching and mixing sequence as it will be added on the job. Proportion the concrete mixtures to have the cement content specified for use in the work. If the maximum size of coarse aggregate is greater than 1 in. [25.0 mm], screen the concrete over a 1-in. [25.0-mm] sieve prior to fabricating the test specimens.

11.4.1 *Other Use Conditions*—Other conditions are known to affect the overall suitability of the concrete mixture for specific intended uses. These include the temperature of the materials or the surroundings, the humidity, the length of time

between mixing and placing, the amount of mixing activity and other factors. These physical conditions may be incorporated into the tests with intention for indicating the potential interactions. These tests would be only for guidance. After incorporation of such test conditions it would not be suitable to expect compliance with this specification requirement.

11.5 *Preparation and Batching*—Prepare all material and make all weighings as prescribed in Practice C 192/C 192M.

**12. Proportioning of Concrete Mixtures**

12.1 *Proportions*—Except when tests are being made for specific uses, all concrete shall be proportioned using ACI 211.1–91 to conform to the requirements described in 12.1.1–12.1.4. After evaluation of the trial mixtures, aggregate proportions shall be adjusted as needed to obtain workable, cohesive mixtures with the correct yield to obtain the required contents. Unless otherwise specified, the admixture shall be added with the first increment of mixing water that is added to the mixer.

12.1.1 The cement content shall be  $517 \pm 5 \text{ lb/yd}^3$  [ $307 \pm 3 \text{ kg/m}^3$ ].

12.1.2 For the first trial mixture, refer to the table on volume of coarse aggregate per unit volume of concrete in ACI 211.1–91 for guidance on the amount of coarse aggregate to use, given the nominal maximum size of the aggregate and the fineness modulus of the fine aggregate being used (see Note 6

NOTE 6—Values in the referenced table of ACI 211.1–91 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 this table be increased by about 7 for the first trial mixture.

12.1.3 For the non-air-entrained mixtures, the air content used in calculating the proportions shall be 1.5, as shown in Table number 5.3.3 of ACI 211.1–91. For the air-entrained mixtures, the air content used for this purpose shall be 5.5.

12.1.4 Adjust the water content to obtain a slump of  $3\frac{1}{2} \pm \frac{1}{2} \text{ in.}$  [ $90 \pm 15 \text{ mm}$ ]. 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. Achieve these conditions by final adjustments in the proportion of fine aggregate to total aggregate or in the amount of total aggregate, or both, while maintaining the yield and slump in the required ranges.

12.2 *Conditions*—Prepare concrete mixtures both with and without the admixture under test. Refer herein to the concrete mixture without the chemical admixture as the reference or control concrete mixture. Add the admixture in the manner recommended by the manufacturer and in the amount necessary to comply with the applicable requirements of the specifications for water reduction or time of setting, or both. When desired by the person or agency for whom the tests are being performed, the admixture is allowed to be added in an amount such as to produce a specific time of setting of the concrete mixture within the limits of the applicable provisions of this specification.

12.2.1 *Non-Air-Entrained Concrete*—When the admixture is to be tested for use only in non-air-entrained concrete, the air content of both the mixture containing the admixture under test and the reference concrete mixture shall be 3.5 % or less, and

<sup>7</sup> Vinsol resin is manufactured by Hercules Inc., Wilmington, DE.

the difference between the air contents of the two mixtures shall not exceed 1.0. If necessary, the air-entraining admixture shall be added to the reference concrete mixture. Tests for resistance to freezing and thawing shall not be made.

12.2.2 *Air-Entrained Concrete*—When the admixture is to be tested for use only in air-entrained concrete, the air-entraining admixture shall be added to the reference concrete mixture and, if necessary, to the concrete mixture containing the admixture under test in sufficient amounts to produce air contents in the range 3.5 to 7.0 %, except that for tests for resistance to freezing and thawing, the range shall be  $6.0 \pm 1.0$  %. In both cases the difference between the air content of the reference concrete and that of the concrete containing the admixture under test shall not exceed 0.5.

**13. Mixing**

13.1 Machine mix the concrete as prescribed in Practice C 192/C 192M.

**14. Tests and Properties of Freshly Mixed Concrete**

14.1 Samples of freshly mixed concrete from at least three separate batches for each condition of concrete shall be tested in accordance with the methods described in 14.2-14.5.

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

14.3 *Air Content*—Test Method C 231.

14.4 *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  $73 \pm 3^\circ\text{F}$  [ $23.0 \pm 2.0^\circ\text{C}$ ].

14.5 *Water Content*:

14.5.1 Report the water-cement ratio of the concrete, computed to the nearest 0.001, as follows: Determine the net water content of the batch as the weight of water in the batch in excess of that present as absorbed water in the aggregates. Calculate the actual volume of concrete in the batch by determining the density of concrete in the batch as prescribed in Test Method C 138. Determine the water-cement ratio by dividing the net weight of water by the weight of cement in the batch.

14.5.2 Calculate the relative water content of the concrete containing the admixture under test as a percentage of the water content of the reference concrete as follows: Divide the average water content of all batches of concrete containing the admixture under test by the average water content of all batches of the reference concrete and multiply the quotient by 100.

**15. Preparation of Test Specimens**

15.1 Make specimens for tests of hardened concrete, representing each test and age of test and each condition of concrete being compared, from at least three separate batches, and the minimum number of specimens shall be as prescribed in Table 2. On a given day make at least one specimen for each test and age of test from each condition of concrete, except make at least two specimens for the freezing and thawing test from each condition of concrete. If desired, the preparation of all specimens can be completed in one, two, or three days of mixing, provided the test concrete and its reference are made on the same day.

**TABLE 2 Types and Minimum Number of Specimens and Tests**

	Num-ber of Types of Specimens <sup>A</sup>	Num-ber of Test Ages	Number of Con-ditions of Con-crete <sup>B</sup>	Num-ber of Speci-mens, min
Water content	...	1	2	<sup>C</sup>
Slump	1	1	2	C
Air content	1	1	2	C
Time of setting	1	<sup>D</sup>	2	6
Compressive strength	1	5	2	30
Flexural strength	1	3	2	18
Freezing and thawing	1	1	2	12
Length change	1	1	2	6
Water reducing, high range	...	6	...	36
Water reducing, high —range and retarding	...	6	...	36

<sup>A</sup> See Section 14 and 16.2.

<sup>B</sup> See 12.2.

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

<sup>D</sup> See 14.4.

15.2 *Manifestly Faulty Specimens*—Visually examine each group of specimens representing a given test or a given age of test, including tests of freshly mixed concrete, 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.

**16. Test Specimens of Hardened Concrete**

16.1 *Number of Specimens*—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 2 shall be made for each condition of concrete to be compared.

16.2 *Types of Specimens*—Specimens made from concrete with and without the chemical admixture under test shall be prepared in accordance with the following:

16.2.1 *Compressive Strength*—Make and cure test specimens in accordance with Practice C 192/C 192M.

16.2.2 *Flexural Strength*—Make and cure test specimens in accordance with Practice C 192/C 192M.

16.2.3 *Resistance to Freezing and Thawing*— Test specimens shall consist of prisms made and cured in accordance with the applicable requirements of Practice 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 chemical admixture under test and from the reference concrete mixture, the air content of each mixture being as specified in 12.2.2.

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

## 17. Tests on Hardened Concrete

17.1 Test specimens of hardened concrete (see Table 1) in accordance with the following methods :

17.1.1 *Compressive Strength*—Test Method C 39/C 39M. Test specimens at ages of 1, 3, 7, and 28 days, 6 months, and 1 year. 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:

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

17.1.1.2 When tests are conducted with materials representative of those proposed for a specific use in accordance with 11.4, and if the results of the tests are required in a period of time that will not permit curing of specimens to ages of 6 months and 1 year, the tests at those ages are permitted to be waived.

17.1.2 *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:

17.1.2.1 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.

17.1.3 *Resistance to Freezing and Thawing*—Comparison tests of the concrete containing the admixture under test with the reference concrete mixture shall be made concurrently using Procedure A of Test Method C 666. Place specimens under test at the age of 14 days. Calculate the relative durability factors as shown in Specification C 260.

17.1.4 *Length Change*—Test specimens shall consist of molded prisms made and tested in accordance with Test Method C 157/C 157M except that the moist curing period, including the period in the molds, shall be 14 days. Then store the specimens in air under conditions specified in the section on Air Storage of Test Method C 157/C 157M for a period of 14 days, at which time determine the length change of the specimen. Consider the drying shrinkage to be the length change during the drying period, based on an initial measurement at the time of removal of the specimen from the mold, and express it as percent to the nearest 0.001 % based on the specimen gage length. If the length change of the reference concrete after 14 days of drying is 0.030 % or greater, the length change on drying of concrete containing the admixture under test, expressed as percent of the length change of the reference concrete, shall not exceed the maximum specified in Table 1. If the length change of the reference concrete after 14 days of drying is less than 0.030 %, the length change on drying of concrete containing the admixture under test shall be not more than 0.010 percentage units greater than that of the reference concrete.

NOTE 7—Since the specific effects produced by chemical admixtures may vary with the properties of the other ingredients of the concrete,

results of length change tests using aggregates of such a nature that the length change on drying is low may not accurately indicate relative performance to be expected with other aggregates having properties such as to produce concrete of high length change on drying.

## 18. Uniformity and Equivalence Tests

18.1 *Infrared Analysis*—This test procedure is intended to compare qualitatively the composition of different samples and results should not be interpreted quantitatively. Sections 18.1.1, 18.1.2, and 18.1.3 give a general procedure for the infrared analysis of admixtures (see Note 8).

18.1.1 *Liquid Admixtures*—Determine the dissolved solids concentration by 18.2 and dilute an aliquot of the liquid admixture sample with distilled water to yield a dissolved solids concentration of about 0.015 g/mL, for example, a 5-mL aliquot diluted to 200 mL. Pipet 5 mL of above solution and add it to a petri dish with 2.5 g of potassium bromide of a grade suitable for use in infrared analysis and 5 mL of distilled water. Stir and mix to dissolve. Place in a drying oven (18.2.1.1) and dry for  $17 \pm \frac{1}{4}$  h at  $105 \pm 3^\circ\text{C}$ . Cool and transfer the dried residue to a mortar and grind to a fine powder. Work quickly to avoid moisture pick-up. Weigh 0.1 g of the powder and 0.4 g of potassium bromide of a grade suitable for use in infrared analysis. Mix in an electric amalgamator for 30 s using stainless steel capsule and balls. Proceed in accordance with 18.1.3.

18.1.2 *Non-liquid Admixtures*—Grind 10 g to a fine powder with mortar and pestle. Transfer the sample to a petri dish, place in a drying oven (18.2.1.1) and dry for  $17 \pm \frac{1}{4}$  h at  $105 \pm 3^\circ\text{C}$ . Weigh approximately 0.005 g of the dry powder and 0.995 g of potassium bromide of a grade suitable for use in infrared analysis. Mix in an electric amalgamator for 30 s using stainless steel capsule and balls. Proceed in accordance with 18.1.3.

18.1.3 To prepare a disk for infrared analysis, weigh 0.300 g of the mixture prepared in 18.1.1 or 18.1.2 and transfer into a suitable die. If an evacuable die is used, apply vacuum for 2 min prior to pressing. Continue vacuum and press at a suitable force for 3 min, producing a disk about 1 mm thick. Remove the disk from the die, insert into the infrared spectrophotometer and obtain infrared absorption spectra.

NOTE 8—It is important that the same procedures be used on all samples to be compared with each other and preferably that they be conducted by the same analyst. Major changes in infrared spectra may result from (a) water content differences due to drying variations, (b) water picked up by hygroscopic materials, (c) reaction between the potassium bromide and some other compound present, and (d) differences in time between formation of the disk and its use. Also, the threshold for detection of individual components by infrared absorption varies widely, depending upon the identity and concentration of accompanying substances. For example, significant amounts of saccharides may be present in a lignosulfonate admixture without their presence being indicated by this method.

18.2 *Residue by Oven Drying (Liquid Admixtures)*:

18.2.1 Place 25 to 30 g of standard Ottawa sand (20 to 30 mesh) in a wide-mouth, low-form (about 60 mm inside diameter and 30 mm in height) glass weighing bottle provided with a ground-glass stopper. Place the weighing bottle and stopper, with stopper removed, in a drying oven (18.2.1.1) and dry for  $17 \pm \frac{1}{4}$  h at  $105 \pm 3^\circ\text{C}$  (Note 7). Insert the stopper in



the weighing bottle, transfer to a desiccator, cool to room temperature, and weigh to the nearest 0.001 g. Remove the stopper and, using a pipet, evenly distribute 4 ml of the liquid admixture over the sand. Immediately insert the stopper to avoid loss by evaporation and weigh to the nearest 0.001 g. Remove the stopper and place both the bottle and stopper in a drying oven (18.2.1.1). Dry for  $17 \pm \frac{1}{4}$  h at  $105 \pm 3^\circ\text{C}$ . At the end of the drying period, stopper the weighing bottle, transfer to a desiccator, cool to room temperature, and weigh to the nearest 0.001 g.

18.2.1.1 *Drying Oven*—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.

18.2.2 *Calculation:*

18.2.2.1 Record the following masses:

- $m_1$  = mass of stoppered bottle with sand and sample,
- $m_2$  = mass of stoppered bottle with sand,
- $m_3$  =  $m_1 - m_2$  = mass of sample,
- $m_4$  = mass of stoppered bottle with sand and dried residue,  
and
- $m_5$  =  $m_4 - m_2$  = mass of dried residue.

18.2.2.2 Calculate the residue by using the following equation:

$$\text{Residue by oven drying (percent by mass)} = (m_5 \times 100)/m_3 \quad (1)$$

NOTE 9—For laboratories conducting this test as a routine operation, previously dried sand and weighing bottles can be maintained in desiccators so that they are immediately available for use when a sample is to be tested.

18.2.3 *Precision Statement*—The maximum multilaboratory coefficient of variation for residue by oven drying (liquid admixtures) has been found to be 1.25 %. Therefore, results of tests by two different laboratories on identical samples of an admixture are not expected to differ from each other by more than 3.5 % of their average (Note 10). The maximum single-operator coefficient of variation has been found to be 0.6 %. Therefore, results of two properly conducted tests by the same operator on the same material are not expected to differ by more than 1.7 %.

NOTE 10—The precision statements are based on the maximum variation of tests made in 18 laboratories on sets of three duplicate samples of two different admixtures.

18.3 *Residue by Oven Drying (Nonliquid Admixtures):*

18.3.1 Place about 3 g of the nonliquid admixture into a dried and tared glass-stoppered weighing bottle (similar to the one described in 18.2.1). Stopper and determine the mass of the bottle and contents to the nearest 0.001 g. Remove the stopper and immediately place both bottle and stopper in a drying oven (18.2.1.1). Dry for  $17 \pm \frac{1}{4}$  h at  $105 \pm 3^\circ\text{C}$ . At the end of the drying period, stopper the weighing bottle, transfer to the desiccator, cool to room temperature, and weigh to the nearest 0.001 g.

18.3.2 *Calculation:*

18.3.2.1 Record the following masses:

- $m_1$  = mass of tared stoppered weighing bottle and sample before drying,
- $m_2$  = mass of empty, stoppered weighing bottle,
- $m_3$  = mass of sample =  $(m_1 - m_2)$ ,
- $m_4$  = mass of tared stoppered weighing bottle and sample after drying, and
- $m_5$  = mass of oven-dried residue =  $m_4 - m_2$ .

18.3.2.2 Calculate the oven-dried residue by using the following equation:

$$\text{Residue by oven drying (mass percent)} = [m \times 100]/m_3 \quad (2)$$

18.3.3 *Precision Statement*—The maximum multilaboratory coefficient of variation for residue by oven-drying (non-liquid admixture) has been found to be 1.40 %. Therefore, results of tests by two different laboratories on identical samples of an admixture are not expected to differ from each other by more than 4.0 % of their average. The maximum single-operator coefficient of variation for residue by oven drying (non-liquid admixture) has been found to be 0.48 %. Therefore, results of two properly conducted tests by the same operator on the same material are not expected to differ by more than 1.4 % of their average. Note 10 also applies to 18.3.3.

18.4 *Specific Gravity (Liquid Admixtures):*

18.4.1 Determine the specific gravity at  $25 \pm 1^\circ\text{C}$  of a liquid admixture using hydrometers complying with Specification E 100. Hydrometers No. 112H through 117H will cover the range for most determinations. A 250-mL graduated cylinder, and a water bath capable of maintaining  $25 \pm 1^\circ\text{C}$  will also be required.

18.4.2 Place a sample in the 250-mL graduated cylinder and put in the hydrometer in such a manner that it floats free and does not touch the side of the cylinder. Place the cylinder with sample and hydrometer in the constant-temperature bath until the temperature of the cylinder, hydrometer, and sample is uniform at  $25 \pm 1^\circ\text{C}$ . If all are at proper temperature prior to insertion of the hydrometer, approximately 10 min should be allowed for equilibrium. If the sample shows evidence of foaming, hydrometer reading should be continued until constant readings are obtained. Read the hydrometer at the base of the meniscus to the nearest 0.005.

18.4.2.1 If foaming is encountered during transfer of the admixture to the cylinder, sufficient time shall be allowed for the foam to dissipate or rise to the surface, where it shall be removed before inserting the hydrometer. Crusting of the admixture on the hydrometer stem due to evaporation during temperature adjustment shall be avoided.

18.4.3 *Precision Statement*—The maximum multilaboratory coefficient of variation for specific gravity (liquid admixtures) has been found to be 0.316 %. Therefore, results of two different laboratories on identical samples of an admixture are not expected to differ from each other by more than 0.9 % of their average (Note 10). The maximum single-operator coefficient of variation has been found to be 0.09 %. Therefore, results of two properly conducted tests by the same operator on the same material are not expected to differ by more than 0.275 %.



**19. Report**

19.1 Report the following:

19.1.1 Results of the tests specified in Sections 6, 14, and 17, and the relevant specification requirements with which they are compared,

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

19.1.3 Brand name, manufacturer's name, and other pertinent data on the material used as the air-entraining admixture,

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

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

19.1.6 Detailed data on the concrete mixtures used, including amounts and proportions of admixtures used, actual cement factors, water-cement ratios, unit water contents, ratios of fine to total aggregate, slump, and air content, and

19.1.7 In the event that, in accordance with the provisions of 17.1.1.2, some of the tests have been waived, the circumstances under which such action was taken shall be stated.

**20. Keywords**

20.1 accelerating; chemical admixtures; concrete; physical requirements; retarding; testing; water reducing

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