Designation: C 1017/C 1017M – 03

Standard Specification for Chemical Admixtures for Use in Producing Flowing Concrete[1]

This standard is issued under the fixed designation C 1017/C 1017M; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

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

1. Scope

1.1 This specification covers two types of chemical admixtures to be added to hydraulic cement concrete mixtures for the purpose of producing flowing concrete. The types are as follows:

1.1.1 *Type I*—Plasticizing, and

1.1.2 *Type II*—Plasticizing and retarding.

1.2 This specification stipulates tests of a chemical admixture with reference concreting materials or with concrete-making materials proposed for specific work. Unless otherwise specified by the purchaser, the tests shall be made using reference concreting materials.

1.3 If a chemical admixture has been tested and found to comply with the provisions of this specification using reference materials, and is being considered for use with other materials for specific work, additional tests for such use are allowed if agreed upon between the purchaser and the supplier and are allowed to consist of a portion of the tests described herein.

1.4 This specification provides for three levels of testing.

1.4.1 *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 the section on Lot Uniformity and Equivalence shall be carried out to provide results against which later comparisons can be made.

1.4.2 *Level 2*—Limited retesting described in the section relating to general requirements may be requested at intervals by the purchaser. Proof of compliance with the requirements of Table 1 demonstrates continued conformity of the admixture with the requirements of the specification.

1.4.3 *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 the section on Lot Uniformity and Equivalence shall be used.

1.5 The values stated in SI units are to be regarded as standard. The values in inch-pound units shown in parentheses are provided for information purposes only.

NOTE 1—It is recommended that, whenever practicable, tests be made using the concrete-making materials (cement, pozzolan, slag, aggregates, air-entraining admixture), the mixture proportions, and the batching sequence and other physical conditions proposed for the specific work. The specific effects produced by chemical admixtures may vary with the properties and proportions of the other ingredients of the concrete.

NOTE 2—Temperature has a pronounced effect on time of setting of concrete. This may be exaggerated by the use of admixture Types I and II. If concrete temperatures to be expected on a particular job differ significantly from the conditions set forth in this specification, further testing may be desirable.

NOTE 3—An unusually rapid loss of workability with time, sometimes termed "slump loss", can be experienced with these admixtures. The rate of slump loss will vary with the particular concreting materials and proportions, mixing equipment and procedures, and temperatures experienced on any particular job. At elevated temperatures, the slump may be retained for a longer period if a Type II admixture is used.

NOTE 4—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 (see ACI 318).

NOTE 5—Admixtures that contain relatively large amounts of alkali ($Na_2O + 0.658\ K_2O$) may contribute to reaction with some aggregates. Compliance with the requirements of this specification does not assure acceptability when used with alkali-reactive aggregates and some cements.

1.6 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.

1.7 The following precautionary caveat pertains only to the test method sections 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*

[1] This specification is under the jurisdiction of ASTM Committee C09 on Concrete and Concrete Aggregates and is the direct responsibility of Subcommittee C09.23 on Chemical Admixtures.

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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:* [2]

C 33 Specification for Concrete Aggregates
C 39 Test Method for Compressive Strength of Cylindrical Concrete Specimens
C 78 Test Method for Flexural Strength of Concrete (Using Simple Beam with Third-Point Loading)
C 136 Test Method for Sieve Analysis of Fine and Coarse Aggregates
C 138/C 138M Test Method for Density (Unit Weight), Yield, and Air Content (Gravimetric) of Concrete
C 143 Test Method for Slump of Hydraulic Cement Concrete
C 150 Specification for Portland Cement
C 157 Test Method for Length Change of Hardened Hydraulic-Cement, Mortar, and Concrete
C 173 Test Method for Air Content of Freshly Mixed Concrete by the Volumetric Method
C 183 Practice for Sampling and the Amount of Testing of Hydraulic Cement
C 192 Practice for Making and Curing Concrete Test Specimens in the Laboratory
C 231 Test Method for Air Content of Freshly Mixed Concrete by the Pressure Method
C 260 Specification for Air-Entraining Admixtures for Concrete
C 403 Test Method for Time of Setting of Concrete Mixtures by Penetration Resistance
C 666 Test Method for Resistance of Concrete to Rapid Freezing and Thawing
C 778 Specification for Standard Sand
D 75 Practice for Sampling Aggregates
D 1193 Specification for Reagent Water
E 100 Specification for ASTM Hydrometers
Manual of Aggregate and Concrete Testing

2.2 *American Concrete Institute Standard:*[3]

ACI 211.1-81 Recommended Practice for Selecting Proportions for Normal and Heavyweight Concrete
ACI 318-83 Building Code Requirements for Reinforced Concrete

3. Terminology

3.1 *Definitions of Terms Specific to This Standard:*

3.1.1 *flowing concrete*—concrete that is characterized by a slump greater than 7½ in. (190 mm) while maintaining a cohesive nature, and which otherwise meets the requirements of Table 1.

TABLE 1 Physical Requirements[A]

	Type I Plasticizing	Type II Plasticizing and Retarding
Time of setting, allowable deviation from Reference, h		
Initial: at least not more than	...	1 later
	1 earlier nor 1½ later	3½ later
Final: at least not more than	...	...
	1 earlier nor 1½ later	3½ later
Increase in slump, min.	90 mm (3.5 in.)	90 mm (3.5 in.)
Compressive strength, min., % of reference		
3 days	90	90
7 days	90	90
28 days	90	90
6 months	90	90
1 year	90	90
Flexural strength, min., % of reference		
3 days	90	90
7 days	90	90
28 days	90	90
Length change after 14 days drying, max. shrinkage (alternative requirements):[B]		
% of reference	135	135
Increase over reference, in.	0.010	0.010
Relative durability factor, min.[C]	80	80

[A] The values in the table include allowance for normal variation in the test results. The object of the 90 % strength requirements is to require a level of performance comparable to that of the reference concrete.

[B] *Alternative Requirements*—Percent of reference limit applies when length change of reference is 0.030 % or greater; increase over reference limit applies when length change of reference is less than 0.030 %.

[C] The 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.

3.1.2 *plasticizing admixture*— a chemical admixture which, when added to concrete, produces flowing concrete without further addition of water and does not retard the setting of the concrete.

3.1.3 *plasticizing and retarding admixture*—a chemical admixture which, when added to concrete, produces flowing concrete without further addition of water and retards the setting of concrete.

3.1.4 *reference concrete*—concrete which contains the same amounts of cement, aggregates, water, and other concreting materials, but no plasticizing admixture.

3.1.5 *test concrete*—concrete which has a plasticizing admixture added.

4. Ordering Information

4.1 When the purchaser specifies flowing concrete, he shall also specify the type of chemical admixture desired. If not specified, the requirements of Type I will apply.

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.

[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.

[3] Available from the American Concrete Institute, 38800 Country Club Drive, Farmington Hills, MI 48331.

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.

NOTE 6—Additional performance tests currently in this standard may be required by users having special requirements.

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 for conformance under this specification.

5.4 At the request of the purchaser, when the chemical 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 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. Lot 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 initial sample and the test sample, obtained as specified, shall be essentially similar.

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

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

6.1.4 *Specific Gravity (Liquid Admixtures)*—When tested as specified, 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. If 10 % of the difference between the specific gravity of the initial sample and water is less than 0.01, use the value 0.01 as the maximum allowable difference. 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. Sampling and Inspection

7.1 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.

7.2 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.

7.3 Samples will be taken for two reasons:

7.3.1 *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.

7.3.2 *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.

7.4 *Liquid Admixtures*—Liquid admixtures shall be agitated thoroughly immediately prior to sampling. Grab samples taken for quality or uniformity tests shall represent not more than 2500 gal (9500 L) of admixture and shall have a volume of at least 1 qt (1 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.

7.4.1 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.

7.4.2 Samples shall be packaged in impermeable, airtight containers which are resistant to corrosion.

7.5 *Nonliquid Admixtures*—Grab samples taken for quality or uniformity tests shall represent not more than 2 tons (1800 kg) of admixture and shall weigh at least 2 lb (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 (2.3 kg) for the composite sample. Grab samples shall be taken from different locations well distributed throughout the quantity to be represented.

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

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

7.6 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.

TEST METHODS

8. Scope

8.1 These test methods are based on arbitrary stipulations which make possible highly standardized testing in the laboratory and are not intended to simulate actual job conditions.

9. Apparatus

9.1 *Infrared Spectrophotometer*.

9.2 *Hydrometers*, Nos. 112H through 117H in accordance with Specification E 100.

9.3 *Water Bath*, capable of maintaining 25 ± 1°C.

10. Reagents and Materials

10.1 *Reagents*:

10.1.1 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean reagent water as defined by Types III or IV of Specification D 1193.

10.1.2 *Potassium Bromide*, of a grade suitable for infrared analysis.

10.2 *Materials*:

10.2.1 *Cement*—The cement used in any series of tests shall be either the cement proposed for specific work or a cement conforming to the requirements of Specification C 150, Type I or Type II. If, when using a cement other than that proposed for specific work, the air content of the concrete made without admixture is more than 3.0 %, select a different cement so that the air content of the concrete will be 3.0 % or less.

10.2.2 *Aggregates*—Except when tests are made using the aggregates proposed for specific work, 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:

10.2.3 *Fine Aggregate*—The fine aggregate grading shall be as follows:

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

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

10.2.4.1 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.

10.2.4.2 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 progress with a scoopful into each succeeding 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 Appendix of Practice D 75, Sampling from Stockpiles, and the Manual of Aggregate and Concrete Testing for guidance for conditions and procedures.

10.2.4.3 Coarse aggregate samples representing each set shall be tested by Test Method C 136 requirements for the sieves shown below. Any set for which the sample does not comply with size 57 shall be discarded. Test results for samples which comply with size 57 shall be averaged for each sieve size. Any set for which the sample deviates from this average by more than the amount shown in column 3 shall be discarded. The process of preparation, testing and averaging shall be continued 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 (1.5 in.)	100	0.0
25.0-mm (1.0 in.)	95 to 100	1.0
12.5-mm (0.5 in.)	25 to 60	4.0
4.75-mm (No. 4)	0 to 10	4.0
2.36-mm (No. 8)	0 to 5	1.0

NOTE 7—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.

10.2.5 *20-30 Sand*—As specified in Specification C 778.

10.2.6 *Air-Entraining Admixture*—Except when tests are made using the air-entraining admixture proposed for specific work, the air-entraining admixture used, if needed in the concrete mixtures, shall be a material which when used to entrain the specified amount of air in the reference 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[4] shall be used.

NOTE 8—If unneutralized Vinsol resin is purchased, neutralization may be accomplished 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 resinate should not exceed 12 to 1 by mass.

10.2.7 *Materials for Tests for Specific Uses*—To test a chemical admixture for use in specific work, the concreting materials 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 25.0 mm (1 in.), screen the concrete over a 25.0-mm (1-in.) sieve prior to fabricating the test specimens.

10.2.7.1 *Other Use Conditions*—At times, other conditions affect the overall suitability of the concrete mixture for specific

[4] Vinsol resin is manufactured by Hercules, Inc., Wilmington, DE.

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 are allowed to 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.

10.2.8 *Preparation and Weighing*—Prepare all material and make all weighings as prescribed in Practice C 192.

11. Proportioning of Concrete Mixtures

11.1 *Proportions*—Except when tests are being made for specific uses, all concrete shall be proportioned using ACI 211.19 to conform to the requirements described below.

NOTE 9—The effects of a chemical admixture on the time of setting and water requirement of concrete may vary with the time of its addition. Consequently, specifications for particular work should require that the admixture be added in the same manner and at the same time as it will be added on the job, or as recommended by the manufacturer, within the restrictions imposed by the specified mixing sequence.

11.1.1 The cement content shall be 335 ± 3 kg/m ^{3}(564 ± 5 lb/yd^3).

11.1.2 For the first trial mixture, refer to the table on volume of coarse aggregate per unit volume of concrete in ACI 211.1 for guidance on the amount of coarse aggregate to use, for the nominal maximum size of aggregate, and for the fineness modulus of the fine aggregate being used (Note 10).

NOTE 10—Values in the referenced table in ACI 211.1 are intended to ensure workable mixtures with the least favorable combinations of aggregate likely to be used.

11.1.3 For the non-air-entrained mixtures, the air content used in calculating the proportions shall be 1.5 %, as shown in Table 5.3.3 of ACI 211.1-81. For the air-entrained mixtures, the air content used for this purpose shall be 5.5 %.

11.1.4 Adjust the water content of both the reference concrete and the test concrete to obtain an initial slump of 90 ± 15 mm (3½ ± ½ in.). The workability of the concrete mixtures shall be suitable for consolidation by hand rodding, and the concrete mixtures 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 range.

11.2 *Conditions*—Prepare batches of the final trial mixture, one of test concrete and one of reference concrete, which are identical prior to addition of the plasticizer. Add the admixture to the test concrete in the manner recommended by the manufacturer and in the amount necessary to obtain a slump of 215 ± 25 mm (8½ ± 1 in.).

11.2.1 *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 test concrete mixture and the reference concrete mixture shall be 3.0 % or less, and the difference between the air contents of the two mixtures shall not exceed 0.5 %. If necessary, the air-entraining admixture shall be added to the reference concrete mixtures.

11.2.2 *Air-Entrained Concrete*—When the admixture is to be tested for use only in air-entrained concrete requiring resistance to freezing and thawing, the air-entraining admixture shall be added to reference and test concrete mixtures to produce air contents in the range of 5.0 to 7.0 %. When entrained air is to be used for workability or other purposes, air contents are adjusted to 3.5 to 7.0 %. In either case, the difference between the air content of the reference concrete and that of the test concrete shall not exceed 0.5 %.

12. Mixing

12.1 Machine mix the reference concrete mixture as prescribed in Practice C 192.

12.2 Machine mix the test concrete mixture as prescribed in Practice C 192, except that the admixture shall be added according to the manufacturer's instructions. In the absence of such instructions, add the admixture at the start of the 2 min final mixing period described in Practice C 192.

13. Tests and Properties of Freshly Mixed Concrete

13.1 Samples of freshly mixed concrete from at least three separate batches for each condition of concrete shall be tested in accordance with the following methods:

13.1.1 *Slump*—Test Method C 143.

13.1.2 *Air Content*—Test Method C 231 or Test Method C 173.

13.1.3 *Time of Setting*—Test Method C 403, 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 ± 2.0°C (73 ± 3°F).

13.1.4 *Water Content and Unit Weight*— 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. Measure the unit weight and compute the yield, cement content, and water content as prescribed in Test Method C 138/C 138M. Determine the water–cement ratio to the nearest 0.01 by dividing the net weight of water by the weight of cement in the batch.

14. Test Specimens of Hardened Concrete

14.1 Make specimens for tests of hardened concrete from at least three separate batches representing each test and age of test from each condition of concrete being compared. 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

TABLE 2 Types and Minimum Number of Specimens and Tests

	Number of Types of Specimens	Number of Test Ages	Number of Conditions of Concrete	Number of Specimens, min.
Slump	1	1	2	[A]
Air content	1	1	2	[A]
Time of setting	1	[B]	2	6
Compressive strength	1	5	2	30
Flexural strength	1	3	2	18
Freezing and thawing	2	1	2	12
Length change	1	1	2	6

[A] Determined on each batch of concrete mixed.
[B] As prescribed in the text.

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. Complete the preparation of all specimens in three days of mixing.

14.2 *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 without testing any specimen found to be manifestly faulty by such examination. Visually examine all specimens representing a given test at a given age after testing. 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.

14.3 *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.

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

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

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

14.4.3 *Resistance to Freezing and Thawing*— Test specimens shall consist of prisms made and cured in accordance with the applicable requirements of Practice C 192. The prisms shall be not less than 75 mm (3 in.) nor more than 130 mm (5 in.) in width and depth, and not less than 275 mm (11 in.) nor more than 410 mm (16 in.) in length. Make one set of specimens from the test concrete mixture and one from the reference concrete mixture, the air content of each mixture being as specified.

14.4.4 *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.

15. Tests on Hardened Concrete

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

15.1.1 *Compressive Strength*—Test Method C 39. Test specimens at ages 3 days, 7 days, 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:

15.1.1.1 Divide the average compressive strength of the specimens made from the test concrete 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. Requirements are shown in Table 1.

NOTE 11—When tests are conducted with materials representative of those proposed for use in specific work, 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 as required in 14.1.1 may be waived.

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

15.1.3 *Resistance to Freezing and Thawing*— Procedure A of Test Method C 666. Place specimens under test at the age of 14 days unless durability at some later age is desired for specific work. Calculate the relative durability factors as shown in Specification C 260. Only air-entrained concrete designed for resistance to freezing and thawing shall be tested for resistance to freezing and thawing.

15.1.4 *Length Change*—Test specimens shall consist of molded prisms made and tested in accordance with Test Method C 157, 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 Test Method C 157 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 14 day 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. Alternative requirements for length change, based on reference concrete length change, are shown in Table 1.

NOTE 12—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 from other aggregates having properties that produce concrete of high length change on drying.

16. Infrared Analysis

16.1 *Scope*—This test method is intended to compare qualitatively the composition of different samples; results should not be not interpreted quantitatively. A general procedure for the infrared analysis of admixtures is given.

NOTE 13—It is important that the same procedures be used on all samples to be compared with each other and it is preferable 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 test method.

16.2 *Procedure*:

16.2.1 *Liquid Admixtures*—Determine the residue by oven drying by 17.1 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 containing 2.5 g of potassium bromide and 5 mL of distilled water. Stir and mix to dissolve. Place in a drying oven and dry for 17 ± ¼ h at 105 ± 3°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. Mix in an electric amalgamator for 30 s using stainless steel capsule and balls. Proceed in accordance with 16.2.3.

16.2.2 *Nonliquid Admixture*—Grind 10 g to a fine powder with mortar and pestle. Transfer the sample to a petri dish, place in a drying oven and dry for 17 ± ¼ h at 105 ± 3°C. Weigh approximately 0.005 g of the dried powder and 0.995 g of potassium bromide. Mix in an electric amalgamator for 30 s using stainless steel capsule and balls. Proceed in accordance with 16.2.3.

16.2.3 To prepare a disk for infrared analysis, weigh 0.300 g of the mixture prepared in 16.2.1 or 16.2.2 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.

17. Residue by Oven Drying

17.1 *Liquid Admixtures*:

17.1.1 Place 25 to 30 g of 20–30 sand (Specification C 778) 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 and dry for 17 ± ¼ h at 105 ± 3°C (Note 14). The drying oven must be either a forced circulation type or one with provision for free access of air. Precise control of temperature and time of drying is essential so that the degree of volatilization of the material other than water from sample will not vary. Transfer to a desiccator, cool to room temperature, insert the stopper in the weighing bottle, 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. Dry for 17 ± ¼ h at 105 ± 3°C. At the end of the drying period, transfer to a desiccator, cool to room temperature, stopper the weighing bottle, and weigh to the nearest 0.001 g.

NOTE 14—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.

17.1.2 *Calculation*:

17.1.2.1 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.

17.1.2.2 Calculate the residue by using the following equation.

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

17.1.3 *Precision*—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 15). 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 15—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.

17.2 *Nonliquid Admixtures*:

17.2.1 Place about 3 g of the nonliquid admixture into a dried and tared glass-stoppered weighing bottle. Stopper and weigh bottle and contents to the nearest 0.001 g. Remove the stopper and immediately place both bottle and stopper in a drying oven. Dry for 17 ± ¼ h at 105 ± 3°C. At the end of the drying period, transfer to the desiccator, cool to room temperature, stopper the weighing bottle, and weigh to the nearest 0.001 g.

17.2.2 *Calculation*:

17.2.2.1 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 = $(M_1 - M_2)$ = mass of sample,
M_4 = mass of tared stoppered weighing bottle and sample after drying, and
M_5 = $(M_4 - M_2)$ = mass of oven-dried residue.

17.2.2.2 Calculate the oven dried residue by using the following equation:

$$\text{Residue by oven drying (mass percent)} = (M_5 \times 100)/M_3 \quad (2)$$

17.2.3 *Precision*—The maximum multilaboratory coefficient of variation for residue by oven drying (nonliquid admixtures) 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 (nonliquid 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 15).

18. Specific Gravity (Liquid Admixtures)

18.1 Determine the specific gravity at 25 ± 1°C of a liquid admixture using hydrometers complying with Specification E 100. Hydrometers Nos. 112H through 117H will cover the range for most determinations. A250-mL graduated cylinder, and a water bath capable of maintaining 25 ± 1°C will also be required.

18.2 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 ± 1°C. If all are at the proper temperature prior to insertion of the hydrometer, approximately 10 min shall be allowed for equilibrium. If the sample shows evidence of foaming, hydrometer reading shall be continued until constant readings are obtained. Read the hydrometer at the base of the meniscus to the nearest 0.005.

NOTE 16—If foaming is encountered during transfer of the admixture to the cylinder, sufficient time should be allowed for the foam to dissipate or rise to the surface, or both, so that it will be removed before inserting the hydrometer. Crusting of the admixture on the hydrometer stem due to evaporation during temperature adjustment should be avoided.

18.3 *Precision*—The maximum multilaboratory coefficient of variation for specific gravity (liquid admixtures) has been found to be 0.316 %. 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 0.9 % of their average (Note 15). 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. Rejection

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

19.2 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.

19.3 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 if requested by the purchaser and is allowed to be rejected if it fails to conform to any of the applicable requirements of this specification.

19.4 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.

19.5 The admixture shall be rejected when the purchaser desires if the test concrete containing it has an air content greater than 3.0 % in non-air-entraining concrete; when the admixture is to be used in air-entrained concrete, it shall be rejected when the purchaser desires if the test concrete containing it has an air content greater than 7.0 %. Except when testing in accordance with 10.2.4, other concrete materials are allowed to be chosen for reference and test concretes in order to conform to these requirements.

20. Certification and Report

20.1 When specified in the purchase order or contract, the manufacturer's or supplier's certification shall be furnished to the purchaser stating that samples representing each lot have been manufactured, tested, and inspected in accordance with this specification and the requirements have been met. When specified in the purchase order or contract, a report of the test results shall be furnished.

20.2 Report the following:

20.2.1 Results of the specified tests and the relevant specification requirements with which they are compared,

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

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

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

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

20.2.6 Detailed data on the concrete mixtures used, including amounts and proportions of admixtures used, actual cement contents, water-cement ratios, densities (unit weights), ratios of fine to total aggregate, slumps, and air contents, and

20.2.7 In the event that, in accordance with the provisions of Note 11, some of the tests have been waived, the circumstances under which such action was taken shall be stated.

21. Packaging and Package Marking

21.1 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 documents accompanying packages or bulk shipments of admixtures.

22. Storage

22.1 The admixture shall be stored in such a manner as to permit easy access for proper inspection and identification of each shipment and adequate protection for the admixture.

23. Keywords

23.1 admixture; concrete; flowing concrete; plasticizing admixtures; plasticizing and retarding admixtures; water-reducing admixtures; water-reducing and retarding admixtures