

Designation: C 465 – 99 (Reapproved 2005)

Standard Specification for Processing Additions for Use in the Manufacture of Hydraulic Cements¹

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1. Scope

1.1 This specification pertains to the criteria and tests to be used for determining whether a processing addition, when used in the recommended amount at the option of the cement producer in the manufacture of hydraulic cements, meets the requirements as prescribed by definition in Specifications C 150, C 1157, C 845, and C 595. The materials listed in the following former ASTM Specifications shall be considered as meeting the requirements of this specification:

C 150 - 62, for Portland Cement²

C 205 - 58 T, for Portland Blast-Furnace Slag Cement³

C 340 - 58 T, for Portland Pozzolan Cement³

C 358 - 58, for Slag Cement³

1.2 The following safety hazards caveat pertains only to the test methods described in 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: 4
- C 33 Specification for Concrete Aggregates
- C 39/C 39M 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 109/C 109M Test Method for Compressive Strength of Hydraulic Cement Mortars (Using 2-in. or [50-mm] Cube Specimens)

- C 114 Test Methods for Chemical Analysis of Hydraulic Cement
- C 115 Test Method for Fineness of Portland Cement by the Turbidimeter
- 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 Con-
- C 150 Specification for Portland Cement
- C 151 Test Method for Autoclave Expansion of Portland Cement
- C 173/C 173M Test Method for Air Content of Freshly Mixed Concrete by the Volumetric Method
- C 185 Test Method for Air Content of Hydraulic Cement Mortar
- C 187 Test Method for Normal Consistency of Hydraulic Cement
- C 191 Test Method for Time of Setting of Hydraulic Cement by Vicat Needle
- C 192 Practice for Making and Curing Concrete Test Specimens in the Laboratory
- C 204 Test Method for Fineness of Hydraulic Cement by Air Permeability Apparatus
- C 205 58T Specification for Portland Blast–Furnace Slag Cement
- C 226 Specification for Air-Entraining Additions for Use in the Manufacture of Air-Entraining Hydraulic Cement
- C 231 Test Method for Air Content of Freshly Mixed Concrete by the Pressure Method
- C 293 Test Method for Flexural Strength of Concrete (Using Simple Beam with Center-Point Loading)
- C 340 58T Specification for Portland Pozzolan Cement
- C 358 58 Specification for Slag Cement
- C 595 Specification for Blended Hydraulic Cements
- C 596 Test Method for Drying Shrinkage of Mortar Containing Portland Cement
- C 617 Practice for Capping Cylindrical Concrete Specimens
- C 845 Specification for Expansive Hydraulic Cement
- C 1157 Performance Specification for Hydraulic Cement
- D 891 Test Methods for Specific Gravity, Apparent, of Liquid Industrial Chemicals

 $^{^{\}rm 1}\,\rm This$ specification is under the jurisdiction of ASTM Committee C01 on Cement and is the direct responsibility of Subcommittee C01.20 on Additions.

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² See 1961 Book of ASTM Standards, Part 4.

³ Discontinued, see 1958 Book of ASTM Standards, Part 4.

⁴ 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.

E 203 Test Method for Water Using Volumetric Karl Fischer Titration

3. Materials

- 3.1 Cements:
- 3.1.1 In cases where it is desired that the proposed addition be accepted for general use in portland cement, tests shall be made on cements prepared from at least five different clinkers. As a minimum, these clinkers shall represent two Type I cements containing not less than 9.0 % C_3A , one Type II cement, and two Type III cements, all conforming to Specification C 150.
- 3.1.2 In cases where it is also desired that the proposed addition be used in blended cements, the test and test procedures shall be as specified with a control and an addition for cement conforming to the appropriate Specification C 595 or C 1157.
- 3.1.3 Processing additions which have been shown to meet the requirements of this specification may also be used in cements conforming to Specification C 845. Testing of the addition with these special cements, where desired, shall be done using the tests and test procedures as specified with a control cement and a cement containing the addition, both conforming to Specification C 845.
- 3.1.4 In cases where it is desired that the proposed addition be limited in use to specific types of cement less in number than required in 3.1.1, the tests and test procedures shall be as specified, and at least two pairs of cements shall be prepared from two clinkers from different plants for each type under specific consideration.
- 3.1.5 In cases where it is desired that the proposed addition be limited in use to a single plant, the tests and test procedures shall be as specified and at least two pairs of cements shall be prepared from clinker representing each type under specific consideration.
- 3.1.6 The two companion cements to be made from any one clinker shall be ground to the same fineness within 7 m²/kg when tested in accordance with Test Method C 115 or within 13 m²/kg when tested in accordance with Test Method C 204, and the SO_3 content, expressed as a percentage of the cement mass and reported to the nearest 0.01 %, shall differ by not more than 0.3, so as to afford comparable samples for indicating the effect of the addition on the cement. Each control cement shall comply with all requirements in the specification applicable to that type of cement, and shall not contain the proposed addition when tested by the method furnished by the producer or seller of the addition.
- 3.1.7 The percentage of each of the following shall be determined for each lot of cement tested: silicon dioxide (SiO₂), aluminum oxide (Al₂O₃), ferric oxide (Fe₂O₃), calcium oxide (CaO), magnesium oxide (MgO), sulfur trioxide (SO₃), ignition loss, insoluble residue, sodium oxide (Na₂O), and potassium oxide (K₂O). There shall also be calculated the potential percentages of the following compounds: tricalcium silicate, dicalcium silicate, tricalcium aluminate, and tetracalcium aluminoferrite. Determinations for the percentage of the addition shall be made, both on the control cements and on those with which the addition was interground, using the method proposed therefore by the sponsor.

3.2 Aggregates—The fine and coarse aggregates shall comply with Specification C 33; the coarse aggregate shall comply with the grading requirements for Size No. 57 or Size No. 67. A sufficient quantity from a single lot of coarse aggregate and from a single lot of fine aggregate shall be provided to complete all tests. To prevent the segregation of particle sizes in the fine aggregate, a single lot of sand sufficient for all tests shall either (I) be separated on the 4.75-mm (No. 4), 1.18-mm (No. 16), 300 μ m (No. 50), and 150 μ m (No. 100) sieves and then be recombined in the required quantity for each batch; or (2) be blended while in a damp condition, and maintained in that condition for the duration of the tests. Under option (2), lots of appropriate size for single mortar and concrete batches shall be carefully split or quartered from the entire batch.

4. General Requirements

- 4.1 Processing additions shall conform to the respective requirements in this specification.
- 4.2 The trade name, source, character of the material, and means for the quantitative determination of the addition in the finished cement shall be furnished by the sponsor, manufacturer, or supplier of the addition, and the information shall form a part of the record of tests of the addition. If the processing addition is a liquid, the specific gravity and percent water content shall also be part of the record.
- 4.2.1 The specific gravity, run in accordance with 7.1.1 shall be within ± 0.05 units of the value reported in 4.2.
- 4.3 Processing additions shall be evaluated by comparing cements containing the addition to otherwise identical cements from the same source without the addition, or containing a processing addition which has been shown to comply with this specification using control cements without any additions, hereinafter designated the "control" cement.
- 4.4 The amount of the processing addition to be interground with the cement for evaluation purposes shall be determined by the sponsor of the addition.
- 4.4.1 The amount of the addition in the cement containing the addition and showing compliance with the requirements of this specification shall be determined quantitatively by means of the quantitative determination required by 4.2.
- 4.4.2 The amount of addition, so determined, shall be used to state the amount of addition that shows compliance with this specification.
- 4.4.3 When tests on cements containing the addition show compliance with the requirements of this specification, the addition in cement may be used in any amount up to the maximum amount showing compliance.
- 4.5 The cement produced for evaluation purposes with the processing addition shall comply with the appropriate Specifications C 150, C 845, C 1157M, or C 595, except that it contains the addition under test. The effect of the addition on the properties of the cement shall also be within the following limits:
- 4.5.1 The percentage of water by mass of cement required for normal consistency of cement containing the addition shall not exceed that required by the corresponding control cement by more than 1.0. For those cements not limited to a fixed water requirement, the percentage of water by mass of cement required for standard consistency of the mortar used for

strength determinations as described in 4.5.4 shall not be increased by more than 2.0 by the addition over that required for the control cement.

- 4.5.2 The time of setting of cement containing the addition shall not vary from the time of setting of the corresponding control cement by more than 1 h or 50 %, whichever is the lesser.
- 4.5.3 The autoclave expansion of cement containing the addition, expressed as a percentage change in length, shall be not more than 0.10 greater than that of the corresponding control cement
- 4.5.4 The compressive strength of mortar cubes made with cement containing the addition, in accordance with Test Method C 109/C 109M, and tested at 1, 3, 7, and 28 days for all types, shall be compared with strengths obtained with the control cement at similar ages. The grand average of these individual strength percentages shall be not less than 95 % of the control cement values. It is required that cubes for companion cements be made and tested on the same days, with storage of specimens side by side in the same section of the moist cabinet during the 24-h curing period. Retesting of companion cements on the same, or a following, day is required in order to provide six, rather than three, test specimens for each cement and age of test.
- 4.5.5 The ultimate drying shrinkage (percent) of mortar made with cement containing the addition shall not be more than 0.025 greater than that of similar mortar made with the corresponding control cement when tested in accordance with Test Method C 596.
- 4.5.6 The compressive strength of the concrete made with cement containing the addition shall be compared with strengths obtained with the control cement at similar ages. The grand average of these individual strength percentages shall be not less than 90 % of the values for the control cement.
- 4.5.7 The flexural strength of concrete made with cement containing the addition shall be compared with strengths obtained with the control cement at similar ages. The grand average of these individual strength percentages shall be not less than 90 % of the values for the control cement.
- 4.5.8 The amount of air-entraining addition required to produce $19 \pm 3 \%$ air in the mortar test made in accordance with Test Method C 185, with the cement containing the addition under test, shall be not greater than 120 % of the amount required to produce, within $\pm 1 \%$, the air content obtained with the control cement. The air-entraining addition used shall meet the requirements of Specification C 226.
- 4.6 Processing additions which provide maximum effects as grinding aids or pack set inhibitors may increase cement flowability to a point where mill retention time is reduced sufficiently to affect significantly the particle size distribution of the resulting cement and its physical-chemical properties. Since mill retention times are controllable by mechanical means in full-scale grinding mills, the true physical-chemical effects of the test additive may, in instances where full-scale tests have shown mill retention time reductions to have significant effects on the properties of the resulting cement, be determined for acceptance purposes by making supplementary laboratory or pilot-mill grinds.

4.7 In the event that the effect of the addition on the properties of cement are determined on the basis of laboratory or pilot mill grinds, this fact shall be entered in the report specified in Section 13, and the specific tests shall be indicated.

5. Sampling Cement

5.1 Samples of the plant-ground cement shall be taken from the product stream during grinding. Prior to the start of sampling a given lot of cement, the mill shall have run for 4 h or long enough to have reached equilibrium under the general conditions that are to govern during the sampling period.

Note 1—Records should be kept as to the rate and continuity of feed of the addition, the form in which the addition is used, strength of solution, magnitude of circulating loads, mill discharge temperature, and feed rate of clinker and gypsum. Product fineness should be determined during the grinding immediately subsequent to sampling.

5.2 As the cement samples are taken, they shall be placed in sealable containers which shall be sealed immediately at the end of the sampling period. Prior to use, the samples of a given lot of cement shall be thoroughly blended to form a uniform, representative composite.

6. Test Methods

6.1 Determine the properties enumerated in this specification in accordance with the test methods prescribed in Sections 7-12.

7. Tests on Liquid Processing Additions

- 7.1 Test the liquid processing addition, in the form normally marketed, in accordance with the following test methods:
- 7.1.1 *Specific Gravity*—Use Test Methods D 891, Test Method A or B.
 - 7.1.2 Water Content—Use Test Method E 203.

8. Tests on Cement

- 8.1 Test cement in accordance with the following standards:
- 8.1.1 *Chemical Analysis of Cement*—Test Methods C 114.
- 8.1.2 Compound Composition—Specification C 150.
- 8.1.3 Fineness of Cement—Test Method C 115 or C 204.
- 8.1.4 *Normal Consistency*—Test Method C 187.
- 8.1.5 *Time of Setting* (*Vicat*)—Test Method C 191.
- 8.1.6 Autoclave Expansion—Test Method C 151.
- 8.1.7 Air Content of Mortar—Test Method C 185.
- 8.1.8 Compressive Strength of Mortar— Test Method C 109/C 109M
- 8.1.9 Drying Shrinkage of Mortar—Test Method C 596.

9. Concrete Mixtures

- 9.1 Preparation and Weighing—Prepare all materials used in making the concrete mixtures and make all weighings as prescribed in Practice C 192. Report the amount of mixing water used in each batch on the basis of saturated, surface-dry aggregates.
- 9.2 Proportions—Design one basic concrete mixture having an actual cement content of $307 \pm 3 \text{ kg/m}^3$ ($517 \pm 5 \text{ lb/yd}^3$), and use in all concrete tests herein specified. Adjust the water content of mixtures to provide concrete having a consistency equal to a 64 ± 13 -mm ($2\frac{1}{2} \pm \frac{1}{2}$ -in.) slump in each case.

Adjust the ratio of fine to coarse aggregate to the optimum for concrete to be consolidated by hand rodding. Recommended trial values for the percentage of fine aggregate in the total aggregate, by absolute volume, are as follows:

Coarse Aggregate, Maximum
25.0 mm (1 in.)

Concrete Without
Entrained Air

Angular
Agounded
45
40

9.3 *Mixing of Concrete*—Mix the concrete in accordance with Practice C 192 except as follows: Hand mixing will not be permitted. The rated capacity of the machine mixer shall not be more than twice the size of the batch used.

10. Tests on Freshly Mixed Concrete

10.1 Test samples of the freshly mixed concrete for slump in accordance with Test Method C 143; unit weight in accordance with Test Method C 138/C 138M; and air content in accordance with Test Method C 138/C 138M, C 231, or C 173/C 173M.

11. Test Specimens of Hardened Concrete

11.1 Number of Specimens—At least three specimens shall be made for each test condition. For each cement containing an addition and its companion control cement, make three rounds of concrete mixed on different days. One round of mixes on a given day shall include both the cement containing the addition and its companion control cement. From each round, make at least one test specimen for each test condition. If necessary, to obtain enough concrete for all test specimens to be made in any one round, it may be necessary to make more than one concrete batch for each round.

11.2 Types of Specimens:

11.2.1 *Compressive Strength*—Compressive strength test specimens shall be cylinders made and cured as prescribed in Practice C 192. Cylinders shall be capped as prescribed in Practice C 617.

11.2.2 *Flexural Strength*—Flexural strength test specimens shall be beams made and cured as prescribed in Practice C 192.

12. Test on Hardened Concrete

- 12.1 Test the specimens on hardened concrete, as specified in Section 11, in accordance with the following methods and at the specified ages:
- 12.1.1 Compressive Strength—Test specimens in accordance with Test Method C 39/C 39M at ages 3, 7, 28 days, and 3 months, except also test Type III cement at 24 h.

12.1.2 Flexural Strength—Test specimens in accordance with Test Method C 293, or Test Method C 78, ages at 3, 7, 28 days, and 3 months, except also test Type III cement at 24 h. By either method of test, turn the specimen on its side with respect to its position as molded and center it on the bearing blocks.

13. Report

- 13.1 The report covering the results of the evaluation of a material proposed for use as a processing addition in the manufacture of portland cement under this specification shall include the following information:
- 13.1.1 Trade name, source and character of the material, and the amount recommended for use, together with means for determination of the proposed addition in the finished cement, all as furnished by the sponsor, manufacturer, or seller of the addition.
- 13.1.2 If the proposed processing addition is a liquid, the specific gravity and percent water content by mass,
- 13.1.3 Detailed results of all analyses and tests prescribed by this specification, and the amount of the addition used, as well as other pertinent information required,
- 13.1.4 Comparison of test results to determine compliance with the requirements prescribed in 4.4,
- 13.1.5 Name and location of the laboratory or laboratories that made the tests covered by the report,
- 13.1.6 Include as an appendix to the report, letters of certification from the various cement manufacturers stating the name of the addition, the amount used, and the type of cement in which used, and
- 13.1.7 The highest amount of addition that has demonstrated compliance with the specification in a particular type of cement (see 4.4.3).

14. Manufacturer's Certification of Liquid Processing Aids

14.1 Upon request of the purchaser in the contract or order, a manufacturer's report shall be furnished at the time of shipment stating the water content and specific gravity of the samples of the material taken during production or transfer and certifying that the specific gravity value is within the specified limits of the originally qualified product and that the product meets the requirements of this Specification.

15. Keywords

15.1 additions; hydraulic cements; processing

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