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Standard Specification for Silica Fume Used in Cementitious Mixtures¹

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1. Scope

1.1 This specification covers silica fume for use in concrete and other systems containing hydraulic cement.

1.2 In the cases of slurried or densified silica fume, perform the tests on the raw silica fume from which these products have been made.

1.3 The units stated in SI are to be regarded as the standard. No other units of measurement are included in this standard.

1.4 The following safety hazards caveat pertains only to the test methods portions, Sections 10 – 19, 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.* Read the material safety data sheets for materials used.

1.5 The text of this standard references notes and footnotes that provide explanatory information. These notes and footnotes (excluding those in tables) shall not be considered as requirements of this standard.

2. Referenced Documents

2.1 *ASTM Standards*:²

C109/C109M Test Method for Compressive Strength of Hydraulic Cement Mortars (Using 2-in. or [50-mm] Cube Specimens)

C114 Test Methods for Chemical Analysis of Hydraulic Cement

C125 Terminology Relating to Concrete and Concrete Aggregates

C135 Test Method for True Specific Gravity of Refractory Materials by Water Immersion

C183 Practice for Sampling and the Amount of Testing of Hydraulic Cement

C185 Test Method for Air Content of Hydraulic Cement Mortar

C219 Terminology Relating to Hydraulic Cement

C311/C311M Test Methods for Sampling and Testing Fly Ash or Natural Pozzolans for Use in Portland-Cement Concrete

C430 Test Method for Fineness of Hydraulic Cement by the 45- μ m (No. 325) Sieve

C441/C441M Test Method for Effectiveness of Pozzolans or Ground Blast-Furnace Slag in Preventing Excessive Expansion of Concrete Due to the Alkali-Silica Reaction

C494/C494M Specification for Chemical Admixtures for Concrete

C604 Test Method for True Specific Gravity of Refractory Materials by Gas-Comparison Pycnometer

C670 Practice for Preparing Precision and Bias Statements for Test Methods for Construction Materials

C1005 Specification for Reference Masses and Devices for Determining Mass and Volume for Use in the Physical Testing of Hydraulic Cements

C1012/C1012M Test Method for Length Change of Hydraulic-Cement Mortars Exposed to a Sulfate Solution

C1069 Test Method for Specific Surface Area of Alumina or Quartz by Nitrogen Adsorption

C1157/C1157M Performance Specification for Hydraulic Cement

C1437 Test Method for Flow of Hydraulic Cement Mortar

3. Terminology

3.1 *Definitions*:

3.1.1 *silica fume, n*—very fine pozzolanic material, composed mostly of amorphous silica produced by electric arc furnaces as a by-product of the production of elemental silicon or ferro-silicon alloys (also known as condensed silica fume and microsilica).

3.1.2 *silica fume, densified, n*—silica fume processed to increase bulk density to facilitate handling and shipping.

3.1.3 *silica fume, undensified, n*—silica fume in its raw, as produced or as collected, unprocessed form.

3.1.4 Other terms in this specification are defined in Terminologies C125 and C219.

¹ This specification is under the jurisdiction of ASTM Committee C09 on Concrete and Concrete Aggregates and is the direct responsibility of Subcommittee C09.24 on Supplementary Cementitious Materials.

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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.

4. Ordering Information

4.1 The purchaser shall specify any optional chemical or physical requirements.

5. Chemical Composition

5.1 Silica fume shall conform to the requirements for chemical composition prescribed in Table 1.

6. Physical Requirements

6.1 Silica fume shall conform to the physical requirements prescribed in Table 2. Optional physical requirements are given in Table 3.

7. Sampling

7.1 When the purchaser desires that the silica fume be sampled and tested to verify compliance with this specification, perform the sampling and testing in accordance with Practice C183, modified as described in 7.3.

NOTE 1—Exercise caution in the interpretation of Practice C183, since there is a difference between the continuous manufacture of hydraulic cement and the generation and collection of silica fume. To a great extent, storage is dictated by the design of the silica-fume collection system. The design of silica-fume collection systems may not have provided for sampling points and practices.

7.2 Practice C183, as modified, is not designed for manufacturing quality control and is not required for manufacturer's certification.

7.3 The following modification of Practice C183 is necessary to render it applicable to silica fume.

7.3.1 Replace the words "hydraulic cement" and "cement" with the words "silica fume" every time that they appear in the text.

7.3.2 All samples, whether grab or composite, shall have a mass of at least 1 kg.

7.3.3 When compliance verification tests of silica fume are required to be made at a laboratory other than that of the silica-fume manufacturer or marketer, coordinate the silica-fume sampling schedule, sample transportation time, and sample testing schedule among the purchaser, manufacturer, and testing laboratory so that the test results will be available when the decision to accept or reject the silica fume must be made.

7.3.4 The section entitled "Sampling" is modified as follows:

7.3.4.1 Take two grab samples or two composite samples for the first 100 Mg of silica fume. Take a grab sample or a composite sample for each subsequent 100 Mg of silica fume, but not less than two samples shall be taken in any sampling program.

7.3.4.2 *From Bulk Storage at Points of Discharge*—Withdraw silica fume from the discharge openings in a steady stream until sampling is completed. In sampling bulk storage at

TABLE 2 Physical Requirements

Oversize:	
Percent retained on 45- μ m (No. 325), max, % ^A	10
Percent retained on 45- μ m (No. 325), max variation from average, percentage points ^B	5
Accelerated pozzolanic strength activity index: ^C	
With portland cement at 7 days, min percent of control	105
Specific surface, min, m ² /g	15

^A Exercise care to avoid retaining agglomerations of extremely fine material.
^B The average shall consist of the ten preceding tests or all of the preceding tests if the number is less than ten.
^C Accelerated pozzolanic strength activity index is not to be considered a measure of the compressive strength of concrete containing the silica fume. This is a measure of the reactivity of a given silica fume with a given cement and may vary with the source of both the silica fume and the cement.

TABLE 3 Optional Physical Requirements^A

Uniformity requirements:	
When air-entraining concrete is specified, the quantity of air-entraining agent required to produce air content of 18.0 vol % of mortar shall not vary from the average established by the ten preceding tests or by all preceding tests if less than ten, by more than, %	20
Reactivity with cement alkalies: ^B	
Reduction of mortar expansion at 14 days, min, %	80
Sulfate resistance expansion, ^C	
(moderate resistance) 6 months, max, %	0.10
(high resistance) 6 months, max, %	0.05
(very high resistance) 1 year, max, %	0.05

^A Will be made only at the request of the purchaser.
^B The indicated tests for reactivity with cement alkalies shall not be requested unless the material is to be used with an aggregate that is regarded as deleteriously reactive with alkalies in hydraulic cement. The test for reduction of mortar expansion may be made using any high-alkali cement in accordance with Test Methods C311/C311M, if the cement to be used in the work is not known or is not available at the time of the test. The test for mortar expansion should be performed by each of the high-alkali cements to be used in the work.
^C Only one limit shall be specified.

points of discharge, while the silica fume is flowing through the openings, take samples at such intervals so that, at a minimum, the sampling requirements of 7.3.4.1 are met.

7.3.5 The section entitled "Amount of Testing" is modified by deleting the first paragraph, "General."

8. Frequency of Tests

8.1 Except for the tests listed in 8.2, make all chemical determinations and physical tests on composite samples representing no more than 400 Mg each. Prepare each composite sample by combining portions from the samples representing each 100 Mg, so that each 100 Mg is represented equally.

8.2 Test for specific surface, density, and accelerated pozzolanic strength activity index using composite samples that represent 3200 Mg or three months of production, whichever gives the highest frequency. Prepare each composite sample by combining portions from the samples representing each 400 Mg or 1 month, whichever gives the highest frequency, so that each sample is represented equally.

9. Preparation of Sample

9.1 Prepare composite samples for tests, as required in Section 8, by arranging all test samples in groups, with each group representing the number of megagrams required by the test or tests for which the composite sample is intended. From

TABLE 1 Chemical Requirements

SiO ₂ , min, %	85.0
Moisture content, max, %	3.0
Loss on ignition, max, %	6.0

each of the samples in a group, take equal portions, sufficient in amount to form a composite sample large enough to permit making the required physical or chemical determinations.

9.2 Prior to testing, mix grab samples and composite samples thoroughly. A clean and dry laboratory concrete drum mixer provides adequate mixing for this purpose. Take care to limit the volume of silica fume in the drum mixer to the range of 10 to 50 % of the drum's total capacity. If necessary, secure a sheet of polyethylene film on the drum with an elastic tiedown to keep the material in the drum. Limit the mixing action to 5 ± 1 min.

9.2.1 When a small sample size precludes the use of a concrete mixer, use a heavy plastic bag, of a capacity at least five times larger than the sample volume, to mix the sample thoroughly. After placing the sample in the bag, close the bag by tying the bag opening tightly, and mix the material by rolling the bag around for 5 ± 1 min.

9.3 Take material for specific tests from a thoroughly mixed sample by using a sampling device (sampling tube, scoop, and so forth) of appropriate size to make a test specimen. Make this test specimen from at least six random subsamples.

TEST METHODS—CHEMICAL ANALYSIS

10. Silicon Dioxide and Total Alkalies

10.1 *Reference Method*—Use the reference method in Test Methods **C114** for cements with insoluble residue greater than 1 %. Analysts performing sodium oxide and potassium oxide determinations shall observe the precautions outlined in the applicable section of Performance Specification **C1157/C1157M** (refer to the section on Test Methods). Most pozzolans dissolve completely in lithium borate fluxes.

11. Moisture Content and Loss on Ignition

11.1 Follow the applicable provisions of Test Methods **C311/C311M**.

TEST METHODS—PHYSICAL TESTS

12. Density

12.1 Determine density using either Test Method **C135** as modified in **12.1.1** or Test Method **C604**.

12.1.1 Test Method **C135** modified as follows:

12.2 *Equipment*:

12.2.1 *Two 500-mL Volumetric Flasks*, Class A.

12.2.2 *Balance*, with an accuracy of at least 0.01 g.

12.2.3 *Constant Temperature Bath*, capable of being regulated within ± 0.5 °C.

12.3 *Deionized Water*:

12.4 *Procedure*:

12.4.1 Determine the density of the material as received, unless otherwise specified, as follows. If density determination on an ignited sample is required, first ignite the sample as described in the test for loss on ignition in the applicable section given in Test Methods **C114**.

12.4.2 Determine the mass (W_p), of a 500-mL volumetric flask, to an accuracy of 0.01 g. Add 30 g of silica fume.

Determine the mass of the flask and the contents (W_a) to the nearest 0.01 g. Add water to the flask to fill it one-half full, and shake it to ensure thorough wetting of the material. Fill to the mark with water. Remove air bubbles by shaking the flask at 15-min intervals until the liquid is free of air or by applying a vacuum to the flask. After all of the air bubbles are removed, place the flask in a constant temperature bath at 23 ± 0.5 °C until the flask and its contents reach a constant temperature. Remove the flask from the water bath; immediately add or remove water, at the same temperature, to the flask to get the meniscus on the mark. Wipe dry the exterior of the flask and determine the mass of the flask and its contents (W_s).

12.4.3 Empty, clean, and determine the mass of the 500-mL volumetric flask, used above, filled to the mark with water (W_t) stabilized at 23 ± 0.5 °C.

12.5 *Calculation*:

$$D_{sf} = \frac{(W_a - W_p)}{500 \text{ mL} - [(W_s - W_a)/D_w]} \quad (1)$$

where:

D_{sf} = density of silica fume, Mg/m³,

W_f = mass of 500-mL volumetric flask, g,

W_a = mass of 500-mL volumetric flask plus approximately 30 g of silica fume, g,

W_s = mass of 500-mL volumetric flask plus silica fume plus water to the mark, g,

W_t = mass of 500-mL volumetric flask plus water to the mark, g, and

D_w = $(W_t - W_f)/500\text{-mL}$, Mg/m³.

12.6 Report the average of two density determinations and the test method used in determining the density.

13. Oversize, Amount Retained When Wet-Sieved on a 45- μm (No. 325) Sieve

13.1 Use Test Method **C430**. Calibrate the sieves in accordance with Test Method **C430**.

NOTE 2—Oversize is used to determine the amount of contaminating material retained on the 45- μm sieve. See **Appendix X2**.

14. Specific Surface

14.1 Determine the specific surface by the BET, nitrogen adsorption method, in accordance with Test Method **C1069**.

15. Air Entrainment of Mortar

15.1 Follow the applicable provisions of Test Methods **C311/C311M**, except use the following test mixture and equation for W_c :

	Test Mixture
Portland cement, g	300
Silica fume, g	30
20–30 Standard Ottawa sand, g	1170
Water, mL, sufficient to give a flow of 80 to 95 %	Y
Neutralized Vinsol resin solution, mL, sufficient to produce an air content of 18 ± 3 %	Z

$$W_c = \frac{300 + 1170 + 30 + (300 \times P \times 0.01)}{300/3.15 + 1170/2.65 + (30/D) + [(300 \times P \times 0.01)/1]} \quad (2)$$

Then calculate:

$$\text{Air content, volume \%} = 100[1 - (W_a/W_c)]W_a = W/400 \quad (3)$$