



Standard Specification for Silica Fume Used in Cementitious Mixtures¹

This standard is issued under the fixed designation C 1240; 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.

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

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:

C 109/C 109M Test Method for Compressive Strength of Hydraulic Cement Mortars (Using 2-in. or [50-mm] Cube Specimens)²

C 114 Test Methods for Chemical Analysis of Hydraulic Cement²

C 125 Terminology Relating to Concrete and Concrete Aggregates³

C 157/C 157M Test Method for Length Change of Hardened Hydraulic-Cement, Mortar, and Concrete³

C 183 Practice for Sampling and the Amount of Testing of Hydraulic Cement²

C 185 Test Method for Air Content of Hydraulic Cement Mortar²

C 219 Terminology Relating to Hydraulic Cement²

C 311 Test Methods for Sampling and Testing Fly Ash or Natural Pozzolans for Use as a Mineral Admixture in Portland-Cement Concrete³

C 430 Test Method for Fineness of Hydraulic Cement by the 45- μ m (No. 325) Sieve²

C 441 Test Method for Effectiveness of Pozzolans or Ground Blast-Furnace Slag in Preventing Excessive Expansion of Concrete Due to the Alkali-Silica Reaction³

C 670 Practice for Preparing Precision and Bias Statements for Test Methods for Construction Materials³

C 1005 Specification for Reference Masses and Devices for Determining Mass and Volume for Use in the Physical Testing of Hydraulic Cements²

C 1012 Test Method for Length Change of Hydraulic-Cement Mortars Exposed to a Sulfate Solution²

C 1069 Test Method for Specific Surface Area of Alumina or Quartz by Nitrogen Adsorption⁴

C 1157 Performance Specification for Hydraulic Cement²

C 1437 Test Method for Flow of Hydraulic Cement Mortar²

3. Terminology

3.1 Definitions:

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

3.1.2 Other terms in this specification are defined in Terminologies C 125 and C 219.

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.

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 and Cementitious Materials.

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² Annual Book of ASTM Standards, Vol 04.01.

³ Annual Book of ASTM Standards, Vol 04.02.

⁴ Annual Book of ASTM Standards, Vol 15.02.

*A Summary of Changes section appears at the end of this standard.

TABLE 1 Chemical Requirements

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

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 C 183, modified as described in 7.3.

NOTE 1—Exercise caution in the interpretation of Practice C 183, 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 C 183, as modified, is not designed for manufacturing quality control and is not required for manufacturer's certification.

7.3 The following modification of Practice C 183 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 (2 lb).

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 (110 tons) of silica fume. Take a grab sample or a composite sample for each subsequent 100 Mg

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 alkalis: ^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 alkalis shall not be requested unless the material is to be used with an aggregate that is regarded as deleteriously reactive with alkalis in hydraulic cement. The test for reduction of mortar expansion may be made using any high-alkali cement in accordance with Test Methods C 311, 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.

(110 tons) 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 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 Make all chemical determinations and physical tests on composite samples representing no more than 400 Mg (440 tons) each. Prepare each composite sample by combining portions from the samples representing each 100 Mg (110 tons), so that each 100 Mg is represented equally.

8.2 Test for specific surface and accelerated pozzolanic strength activity index using composite samples that represent 3200 Mg (3520 tons) or 3 months of production, whichever gives the highest frequency. Prepare each composite sample by combining portions from the samples representing each 400 Mg (440 tons) 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 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 \pm 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, etc.) 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 C 114 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 C 1157 (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 C 311.

TEST METHODS—PHYSICAL TESTS

12. Density

12.1 *Equipment*:

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

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

12.1.3 *Constant Temperature Bath*, capable of being regulated within $\pm 0.5^\circ\text{C}$ (1.0°F).

12.2 *Deionized Water*.

12.3 *Procedure*:

12.3.1 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 C 114.

12.3.2 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 $23 \pm 0.5^\circ\text{C}$ 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).

12.3.3 Empty, clean, and determine the mass of the 500-mL volumetric flask, used above, filled to the mark with water (W_t) stabilized at $23 \pm 0.5^\circ\text{C}$.

12.4 *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/m^3 ,

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/m^3 .

12.5 Report the average of two density determinations.

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

13.1 Use Test Method C 430. Calibrate the sieves in accordance with Test Method C 430.

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 C 1069.

NOTE 3—Manufacturers and examples of nitrogen adsorption instrumentation include Horiba Instruments, Inc., Irvine, CA, 5A-9600; Micromeritics Instrument Corporation, Norcross Georgia, FlowSorb-II 2300; Quantachrome Corporation, Boynton Beach, FL, Quantasorb Jr.; and JUWE Laborgerate Service GmbH, Korschenbroich, Germany, Stroehlein AREA meter II.

15. Air Entrainment of Mortar

15.1 Follow the applicable provisions of Test Methods C 311, 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:

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

where:

W_a = actual mass per unit of volume of mortar as determined by Test Method C 185, g/mL,

W = mass of the specified 400 mL of mortar (see Test Method C 185), g,

W_c = theoretical mass per unit volume, calculated on an air-free basis and using the values for density and quantities of the materials in the mixture, g/mL,

P = percent of mixing water plus Vinsol resin solution based on mass of cement, and

D = density of silica fume used in the mixture, Mg/m^3 .

15.2 Determine the flow in accordance with the applicable provisions of Test Method C 109/C 109M.

16. Accelerated Pozzolanic Strength Activity Index with Portland Cement

16.1 Use the applicable section on strength activity index with portland cement of Test Methods C 311, except change to reflect testing at constant water to cementitious materials ratio. Prepare test specimens from the batch proportions below, molding three cubes from both the control mix and the test mix.

16.1.1 Control Mixture:

- 16.1.1.1 500 g of portland cement,
- 16.1.1.2 1375 g of graded standard sand, and
- 16.1.1.3 242 mL of water.

16.1.2 Test Mixture:

- 16.1.2.1 450 g of portland cement,
- 16.1.2.2 50 g of silica fume,
- 16.1.2.3 1375 g of graded standard sand, and
- 16.1.2.4 242 mL of water.
- 16.1.2.5 *X* grams of dry high-range water reducer, meeting ASTM C 494 Type F, required to produce a flow of 100 to 115 %. Add the high-range water reducer directly to the mixing water in the mixing bowl. Then add the cement or the cement-silica fume mixture and start the mixing cycle.

16.1.3 Determine the flow in accordance with the applicable provisions of Test Method C 1437.

16.1.4 *Storage of Specimens*—After 24 h of initial curing in the moist room ($23 \pm 2^\circ\text{C}$ and relative humidity of not less than 95 %), place the cubes in airtight glass containers and store at $65 \pm 2^\circ\text{C}$ for six days.

16.1.5 Determine the compressive strength, as specified in Test Method C 109/C 109M, of the three specimens of the control mixture and the three specimens of the test mixture at 7 days after molding.

17. Reactivity with Cement Alkalies

17.1 Determine the reduction of mortar expansion in accordance with Test Method C 441, except that the amount of silica fume in the test mixture shall be 10 % by mass of cementitious material.

18. Sulfate Resistance

18.1 Determine sulfate resistance according to Test Method C 1012, except that the amount of silica fume used in the test mixture is 10 % by mass of cementitious material.

19. Bulk Density

19.1 The bulk density of silica fume is defined as the mass of a unit volume of loose silica fume.

19.2 This test method covers determination of the bulk density of silica fume, as silica fume is transferred from one container to another with controlled minimum compaction. Its particular usefulness is in connection with identifying material form (as produced or densified), silo or truck storage capacity, material handling and transportation characteristics.

19.3 Equipment:

19.3.1 *Balance*, meeting Specification C 1005, with a sensitivity of 0.1 g.

19.3.2 *Vibrating Table*⁵, Table top, electromagnetic vibrating table, with a controlled low-amplitude that does not exceed 1 mm linear vibration. Approximate deck size is 175×250 mm with a 5 kg capacity. The amplitude of the vibration shall be capable of being regulated to suit the characteristics of the material being handled.

19.3.3 *Stainless Steel Beaker*, of known volume, not less than 1 L calibrated to the nearest ± 1 mL. Without a spout.

19.4 Procedure:

19.4.1 Determine the mass of the clean dry beaker to the nearest 1 g.

19.4.2 Fill the beaker with silica fume and compact by use of the vibrating table at a mid-range setting for 15 s, adding material as needed.

19.4.3 Screed or strike off the measure, with a straight edge or spatula, to produce a flat, even surface, that is level with rim or edge of the beaker. Wipe off any excess silica fume that may adhere to the sides.

19.4.4 Place the filled measure on the balance and determine the mass of the silica fume to the nearest 1 g.

19.5 Calculation:

19.5.1 Divide the net mass of the silica fume in grams by the volume of the container in milliliters. Multiply by 1000 to express the density in kilograms per cubic meter. To convert the value in kilograms per cubic meter to pounds per cubic foot, divide by 16.01846.

20. Report

20.1 Report the following information:

- 20.1.1 SiO₂ content, %,
- 20.1.2 Moisture content, %,
- 20.1.3 Loss on ignition, %,
- 20.1.4 Oversize, % retained,
- 20.1.5 Bulk density, kg/m³,
- 20.1.6 Density, Mg/m³,
- 20.1.7 Name of manufacturer and brand, if applicable,
- 20.1.8 Accelerated Pozzolanic Strength Activity Index,
- 20.1.9 Specific surface, m²/g, and
- 20.1.10 Available alkalies, as equivalent Na₂O, %.

20.2 Report the following information when specifically requested by the purchaser:

- 20.2.1 The quantity of air-entraining agent compared to the 10 preceding tests, %,
- 20.2.2 Reduction of mortar expansion, %, and
- 20.2.3 Sulfate resistance expansion, %.

21. Precision and Bias

21.1 Precision:

21.1.1 *Accelerated Pozzolanic Strength Activity Index Test:*

21.1.1.1 *Single-Operator Precision*—The precision of this test will be evaluated using Practice C 670.

21.1.1.2 *Multilaboratory Precision*—The precision of this test will be evaluated using Practice C 670.

21.1.2 *Density Test:*

⁵ A suitable vibrating table is the Syntron Paper Jogger, Model J-1, manufactured by F.M.C. Corp., 57 Cooper Ave., Homer City, PA 15748.

21.1.2.1 *Single-Operator Precision*—The single-operator standard deviation among single test results (a test result is defined in this specification as the average of two separate measurements) has been found to be 0.035 Mg/m³.⁶ Therefore, results of two properly conducted tests by the same operator should not differ by more than 0.099 Mg/m³⁶ on the same silica fume.

21.1.2.2 *Multilaboratory Precision*—The multilaboratory standard deviation among single test results (a test result is defined in this specification as the average of two separate measurements) has been found to be 0.047 Mg/m³.⁶ Therefore, results of two properly conducted tests in different laboratories on the same silica fume should not differ by more than 0.132 Mg/m³ of their average.⁶

21.1.3 *Bulk Density*:

21.1.3.1 *Precision*—The precision of this test method will be evaluated using Practice C 670.

21.1.3.2 *Bias*—Since there is no accepted reference material suitable for determining any bias that might be associated with this test method, no statement on bias is being made.

21.2 *Bias*—Since there is no accepted reference material suitable for determining the bias for the procedures for measuring the accelerated pozzolanic strength activity index and the density, no statement on bias is being made.

⁶ These measurements represent, respectively, the (1s) and (d2s) limits in accordance with Practice C 670.

22. Rejection and Retesting

22.1 The purchaser has the right to reject material that fails to conform to the requirements of this specification. Rejection shall be reported to the producer or supplier promptly and in writing. In case of dissatisfaction with the results of the tests, the producer or supplier is not prohibited from making a claim for retesting.

23. Certification

23.1 When specified in the purchase order or contract, the purchaser shall be furnished certification that samples have been tested as directed in this specification and the specified requirements have been met. When specified in the purchase order or contract, a report of the test results shall be furnished.

24. Packaging and Package Marking

24.1 When silica fume is delivered in packages, the name, and brand, if applicable, of the manufacturer or distributor and the mass of the silica fume contained therein shall be marked plainly on each package. Similar information shall be provided in the shipping invoices accompanying the shipment of packaged or bulk silica fume in dry or slurried forms. All packages shall be in good condition at the time of inspection.

25. Storage and Inspection

25.1 Silica fume shall be stored in such a manner as to permit easy access for the proper inspection and identification of each shipment. Facilities for inspection and sampling shall be provided at the point from which the material is to be shipped.

APPENDIXES

(Nonmandatory Information)

X1. SILICA CONTENT

X1.1 Since the quantity of silica in the amorphous state is one of the primary characteristics that determines the amount of activity of silica fume, the chemical analysis for silica content is important. At the present time, there are no National Institute of Standards and Technology (NIST) SRMs of silica fume, and reference silicon dioxides (SiO₂) are therefore the only materials available for instrumental standards. Since silica in this specification is limited to 85 % SiO₂ or higher, silica flour (99.9 %), or silica brick (93.94 %), etc. is adequate for flame atomic absorption (AA) analysis. At the present time, there are problems using inductively coupled plasma (ICP) for the analysis of solutions with high percentages of silica as well

as problems with borate clogging the nebulizer. Another problem is that when a lithium borate fusion is dissolved in HCl, some of the silica returns to a solid phase and can be filtered out. This will affect the total silica analysis by any instrumental method that uses this method of fusion. Energy dispersive X-ray (EDX) is the technique most likely to be used with the X-ray based methods. This technique needs a similar material, a silica fume, for comparison. The “wet method,” sodium carbonate fusion, in which silica is recovered gravimetrically as SiO₂, is the only method, at the present, that requires no standard and yields accurate results.

X2. OVERSIZE

X2.1 The 45- μm (No. 325) sieve specification is to be used to determine the amount of foreign material present. Since silica fume is much finer than cement or fly ash, the particles will all pass through the sieve except for foreign material.

Extremely fine materials tend to form agglomerations; good judgment must be exercised to differentiate between easily dispersible agglomerates and foreign materials.

X3. PROBLEM OF MIXTURE PROPORTIONING FOR VARIOUS TEST MIXTURES

X3.1 Such test methods as accelerated pozzolanic strength activity index with portland cement, reactivity with cement alkalies, and sulfate resistance require mixtures where the silica fume being tested replaces a given amount of cement. For specification purposes, 10 % by mass replacement of cement by silica fume will be used rather than that which is stated in the present methods. Water-to-cementitious materials ratio will be replaced by a flow of between 100 and 115 %. As the percent replacement with silica fume increases, the mixture

becomes unworkable, and either more water is necessary or a water reducer is necessary to have a workable mixture. By limiting the mixtures to 10 % by mass replacement, the addition of water to a certain flow is a viable alternative, even though the addition of water reducer would probably produce a higher strength. Since this is a specification, the interest is in comparing material under similar conditions, rather than in maximum strength.

X4. SULFATE RESISTANCE

X4.1 Satisfactory reductions of expansion in laboratory mixtures have been obtained with silica fume replacement levels of 5 to 15 %. Each source of silica fume must be tested

with high- C_3A portland cement to establish appropriate replacement levels for adequate sulfate resistance.

SUMMARY OF CHANGES

Committee C09 has identified the location of selected changes to this standard since the last issue (C 1240 – 03) that may impact the use of this standard. (Approved Aug. 10, 2003.)

- (1) Section 10 was revised.
- (2) Section 11 was revised.

- (3) Section 2 was revised.
- (4) The title of the standard was revised.

Committee C09 has identified the location of selected changes to this standard since the last issue (C 1240 – 01) that may impact the use of this standard. (Approved Jan. 10, 2003.)

- (1) 8.2 was revised.
- (2) 13.1 was revised.

- (3) 20.1.4 was revised.

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