National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material® 2696

Silica Fume
(powder form)

This Standard Reference Material (SRM) is intended primarily for use in evaluating chemical and instrumental methods of analysis of silica fume used in conjunction with product specifications [1,2]. A unit of SRM 2696 consists of a single bottle containing approximately 70 g of powder.

Certified Mass Fraction Values: Certified values for constituents in SRM 2696 are listed in Table 1 on a dry-mass basis [3]. Value assignment categories are based on the definitions of terms and modes used at NIST for certification of chemical reference materials [4]. A NIST certified value is a value for which NIST has the highest confidence in its accuracy, in that all known or suspected sources of bias have been investigated or taken into account. A certified value is the present best estimate of the true value based on the results of analyses performed at NIST and collaborating laboratories.

Reference Mass Fraction Values: Reference values for constituents on a dry-mass basis [3], loss on ignition at 750 ºC, and specific surface area of SRM 2696 are listed in Table 2. Reference values are noncertified values that represent the best estimates of the true value; however, the values do not meet the NIST criteria for certification and are provided with associated uncertainties that may reflect only measurement precision, may not include all sources of uncertainty, or may reflect a lack of sufficient statistical agreement among multiple analytical methods [4].

Expiration of Certification: The certification of **SRM 2696** is valid, within the measurement uncertainty specified, until **01 May 2023**, provided the SRM is handled and stored in accordance with instructions given in this certificate (see "Instructions for Storage and Use"). This certification is nullified if the SRM is damaged, contaminated, or otherwise modified.

Maintenance of SRM Certification: NIST will monitor this SRM over the period of its certification. If substantive technical changes occur that affect the certification before the expiration of this certificate, NIST will notify the purchaser. Registration (see attached sheet, or register online) will facilitate notification.

Coordination of the technical measurements for certification was accomplished under the direction of J.R. Sieber of the NIST Chemical Sciences Division.

Analytical measurements (including homogeneity testing) for certification of this SRM were performed at NIST by J.R. Sieber and A.F. Marlow of the NIST Chemical Sciences Division.

The material was blended and bottled at NIST under the supervision of M.P. Cronise of the NIST Office of Reference Materials.

Statistical consultation for this SRM was provided by S.D. Leigh of the NIST Statistical Engineering Division.

Support aspects involved in the issuance of this SRM were coordinated through the NIST Office of Reference Materials.

Carlos A. Gonzalez, Chief
Chemical Sciences Division

Gaithersburg, MD 20899
Certificate Issue Date: 08 September 2017
Certificate Revision History on Last Page

Steven J. Choquette, Director
Office of Reference Materials

INSTRUCTIONS FOR STORAGE AND USE

To relate analytical determinations to the certified values on this Certificate of Analysis, a minimum sample quantity of 500 mg is recommended. The material must be dried at 110 ºC for at least 1 h, prior to analysis for chemical constituents. The bottle should be recapped immediately after removal of a sample. Store SRM 2696 in its original container in a cool, dry location, preferably in a desiccator. When a sample is taken from a stored container, it is recommended to determine the loss on ignition and to correct for loss in excess of the reference value, if any. For evaluation of test methods for moisture content and loss on ignition, use only samples taken from a freshly opened pouch.

Warning to Users: In some laboratories, it may be accepted practice to return material to its original container after testing it for Specific Surface Area. Because this SRM is intended for testing of other properties and constituents, the material should be discarded after use. Returning it to the original container may result in contamination of the material and nullification of the certification.

PREPARATION AND ANALYSIS[(1)]

Stability: The silica fume material for SRM 2696 was obtained from Elkem Materials, Inc. from a typical production batch of silica fume. SRM 2696 is considered to be stable during the period of certification when stored in its original bottle in a sealed foil pouch. Once the foil pouch has been opened, the moisture content and loss on ignition may be subject to change (see "Instructions for Storage and Use").

Moisture Content and Loss on Ignition: The reference values were determined by the cooperating laboratories using the methods of test in ASTM C 311-02 [7]. The use of alternative methods may result in different values for these properties. The determinations were made using material from freshly opened units of the SRM.

Specific Surface Area: The reference value for specific surface area provided in Table 2 was determined by the cooperating laboratories using the Brunauer-Emmet-Teller (BET) method of test in ASTM C 1069-86(1997) [8]. The determinations were performed using both single-point and multi-point analyses. The suitability of this SRM for use with different methods and measurement dynamics has not been determined.

Certified Mass Fraction Values: The measurands are the mass fractions of selected constituents in silica fume. The certified values are metrologically traceable to the SI unit of gram per 100 grams. The constituents listed in Table 1 represent consensus assignments of chemical forms by the cement industry and the analytical test methods used by NIST and collaborating laboratories are listed in Table 3.

Table 1. Certified Mass Fraction Values (Dry-Mass basis) for SRM 2696

Constituent	Mass Fraction[(a)] (%)
SiO_2	95.61 ± 0.37
Al_2O_3	0.2080 ± 0.0071
CaO	0.426 ± 0.016
MgO	0.235 ± 0.024[(b)]
K_2O	0.652 ± 0.028
Mn_2O_3	0.032 ± 0.004[(b)]
ZnO	0.051 ± 0.005[(b)]

[(a)] The certified values, unless otherwise footnoted, are weighted means of the mass fractions from two to seven analytical methods [10]. The uncertainty listed with each value is an expanded uncertainty about the mean [10,11], with coverage factor, $k = 2$ (approximately 95 % confidence), calculated by combining a between-source variance incorporating inter-method bias with a pooled within-source variance following the ISO/JCGM Guide [5].

[(b)] The certified values are unweighted means of the mass fractions from two to five analytical methods [10]. The uncertainty listed with each value is an expanded uncertainty about the mean, with coverage factor 2, calculated by combining a between-method variance [11] with a pooled, within-method variance following the ISO/JCGM Guide [5]

[(1)]Certain commercial instruments, materials, or processes are identified in this report to adequately specify the experimental procedure. Such identification does not imply recommendation or endorsement by the National Institute of Standards and Technology, nor does it imply that the instruments, materials, or processes identified are necessarily the best available for the purpose.

Reference Mass Fraction Values: The measurands are the mass fractions of selected constituents in silica fume determined using the test methods listed in Table 3. The measurand for moisture content and loss on ignition is the mass lost at the corresponding temperature using the test methods in ASTM C 311 [7]. The measurand for specific surface area is defined in ASTM method C 1069 [8]. The reference values are metrologically traceable to the SI unit of gram per 100 grams. The constituents listed in Table 2 represent consensus assignments of chemical forms by the cement industry.

Table 2. Reference Values (Dry-Mass Basis) for SRM 2696[(a)]

Constituent	Mass Fraction (%)
Na_2O	0.129 ± 0.019
P_2O_5	0.0863 ± 0.0057[(b)]
Fe_2O_3	0.055 ± 0.011
Moisture Content	0.251 ± 0.034
Loss on Ignition at 750 ºC	2.11 ± 0.10
	Unit (m^2/g)
Specific Surface Area (S)	22.92 ± 0.36

[(a)] The reference values, unless otherwise footnoted, are weighted means from two to seven analytical methods [10]. The uncertainty listed with each value is an expanded uncertainty about the mean [10,11], with coverage factor, $k = 2$ (approximately 95 % confidence), calculated by combining a between-source variance incorporating between-method bias with a pooled within-source variance following the ISO/JCGM Guide [5].

[(b)] The reference value is an unweighted mean of the mass fractions from three analytical methods [10]. The uncertainty listed with each value is an expanded uncertainty about the mean, with coverage factor 2, calculated by combining a between-method variance [11] with a pooled, within-method variance following the ISO/JCGM Guide [5]

Table 3. Analytical Methods[(a)]

Constituent	Methods
Na_2O	XRF, ICP-OES, FAAS, C 114-99
MgO	XRF, ICP-OES, FAAS, C 114-99
Al_2O_3	XRF, ICP-OES, C 114-99
SiO_2	XRF, C 114-99
P_2O_5	XRF, ICP-OES, C 114-99,
K_2O	XRF, ICP-OES, FAAS, C 114-99
CaO	XRF, FAAS, C 114-99
Mn_2O_3	XRF, ICP-OES, C 114-99
Fe_2O_3	XRF, ICP-OES
ZnO	XRF, ICP-OES, C 114-99
Moisture Content	C 311-02
Loss on Ignition at 750 ºC	C 311-02
Specific Surface Area (m^2/g)	C 1069-86(1997), Single-point and Multi-point analyses, N_2 gas

[(a)]**Key:**

XRF: X-ray fluorescence spectrometry with various sample preparation and calibration procedures performed at NIST and collaborating laboratories

ICP-OES: Inductively-Coupled Plasma Optical Emission Spectrometry performed at collaborating laboratories

FAAS: Flame Atomic Absorption Spectrophotometry performed at collaborating laboratories

C 114-99: Classical (wet) chemical methods in this ASTM standard performed at collaborating laboratories

Note: Although C 114-99 is expected to give reliable results for the analysis of silica fume, its application to this matrix has not been validated by ASTM Committee C01.

The Cooperating Laboratories tested SRM 2696 for oversize in accordance with ASTM C 430. The data generated had a wide variance that did not meet NIST criteria for value assignment. In the minutes of the December 5, 2003, meeting of ASTM International Subcommittee C09.24 Task Group 4 on Silica Fume Specification C 1240, the data variance was attributed to the self-agglomerating characteristics of this material and that the measurement of oversize in silica fume requires a modification of ASTM C 430-96(2003) [12].

Cooperating Laboratories: SRM 2696 was produced in cooperation with the Silica Fume Association as Task 11 under Cooperative Agreement DTFH61-99-X00063 between the Federal Highway Administration and the Silica Fume Association. Participation by collaborating laboratories was coordinated by G.M. Gapinski of Norchem, Inc., Ft. Pierce, Florida. Analytical determinations for certification of this SRM were performed by the following laboratories:

G. Gapinski and R. Plotria; Norchem, Inc.; Ft. Pierce, Florida (USA)
K. Groff; New York, Dept. Of Transportation, Materials Bureau; Albany, New York (USA)
J. Beilman; Kansas Dept. of Transportation; Topeka, Kansas (USA)
S. Carlock; Utah Dept. of Transportation, Materials QA Section; Salt Lake City, Utah (USA)
D. Broton; Construction Technology Laboratories; Skokie, Illinois (USA)
R. Martin; Globe Metallurgical, Inc.; Beverly, Ohio (USA)
K. Kasprzak; Globe Metallurgical, Inc.; Niagara Falls, New York (USA)
R. Chevrier; CANMET Materials Technology Laboratory; Ottawa, Ontario (Canada)
R. Hageman; Elkem Materials Inc.; Alloy, West Virginia (USA)
S. Schlorholtz; Iowa State University, Material Analysis Research Laboratory; Ames, Iowa (USA)
R. Karuhn; Particle Technology Labs, Inc.; Downers Grove, Illinois (USA)
M. Thomas; Quantachrome Corp.; Boynton Beach, Florida (USA)
M. Pohl; Horiba Instruments, Inc.; Irvine, California (USA)
R. Xu; Beckman Coulter, Inc., Particle Characterization Operation; Miami, Florida (USA)

REFERENCES

[1] ASTM C 1240-03a; *Standard Specification for Silica Fume Used in Cementitious Mixtures*; Annu. Book ASTM Stand., Vol. 04.02 (2003).

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[7] ASTM C 311-02; *Standard Test Methods for Sampling and Testing Fly Ash or Natural Pozzolans for Use as a Mineral Admixture in Portland-Cement Concrete*; Annu. Book ASTM Stand., Vol. 04.02 (2002).

[8] ASTM C 1069-86(1997); *Standard Test Method for Specific Surface Area of Alumina or Quartz by Nitrogen Adsorption*; Annu. Book ASTM Stand., Vol. 03.05 (1997).

[9] ASTM C 114-99, *Standard Test Methods for Chemical Analysis of Hydraulic Cement*; Annu. Book ASTM Stand., Vol. 04.01 (1999).

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[12] Minutes, ASTM International Subcommittee C09.24 Task Group 4 on Silica Fume Specification C 1240, December 5, 2004.

Certificate Revision History: 08 September 2017 (Title change; editorial changes); 19 December 2013 (Extension of certification period; additional storage information added; editorial changes); 17 September 2007 (Additional information provided about analytical methods; editorial changes); 06 May 2004 (Original certificate date).

Users of this SRM should ensure that the Certificate of Analysis in their possession is current. This can be accomplished by contacting the SRM Program: telephone (301) 975-2200; fax (301) 948-3730; e-mail srminfo@nist.gov; or via the Internet http://www.nist.gov/srm.