National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material® 59a

Ferrosilicon
Grade E1
(powder form)

This Standard Reference Material (SRM) is intended primarily for use in validation of chemical and instrumental methods of analysis for element contents of ferrosilicon and materials of similar matrix. It can be used to validate value assignment of in-house reference materials. A unit of SRM 59a consists of one bottle containing approximately 50 g of powder.

Certified Mass Fraction Values: Certified values for constituents of SRM 59a are reported in Table 1 as mass fractions of the elements in a ferrosilicon matrix [1]. 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 taken into account [2]. A certified value is the present best estimate of the true value. The certified values are metrologically traceable to the SI derived unit of mass fraction (expressed as percent). The expanded uncertainty estimates are expressed at a confidence level of approximately 95 %.

Table 1. Certified Mass Fraction Values for SRM 59a

Constituent	Mass Fraction (%)	Expanded Uncertainty (%)
Aluminum (Al)	0.354	0.011
Boron (B)	0.0578	0.0060
Calcium (Ca)	0.0418	0.0072
Carbon (C)	0.0458	0.0031
Chromium (Cr)	0.0805	0.0039
Copper (Cu)	0.0520	0.0047
Iron (Fe)	50.05	0.15
Manganese (Mn)	0.754	0.012
Nickel (Ni)	0.0328	0.0073
Phosphorus (P)	0.0158	0.0019
Silicon (Si)	48.10	0.30

Expiration of Certification: The certification of **SRM 59a** is valid indefinitely, within the measurement uncertainty specified, provided the SRM is handled and stored in accordance with instructions given in this certificate (see "Instructions for Handling, Storage and Use"). Periodic recertification of this SRM is not required. The 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.

Carlos A. Gonzalez, Chief
Chemical Sciences Division

Gaithersburg, MD 20899
Certificate Issue Date: 11 June 2019
Certificate Revision History on Last Page

Steven J. Choquette, Director
Office of Reference Materials

Coordination of technical measurements for certification was performed by O. Menis and J.I. Schultz, formerly of NIST. Review and revision of values and uncertainty estimates were coordinated by J.R. Sieber of the NIST Chemical Sciences Division.

Statistical consultation for this SRM was provided by A. Possolo of the NIST Statistical Engineering Division.

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

INSTRUCTIONS FOR HANDLING, STORAGE AND USE

Ferrosilicon powder may be analyzed in the as-received form. To relate analytical determinations to the certified values in this Certificate of Analysis, a minimum test portion of 200 mg should be used. Before sampling, it is recommended to mix the powder by inverting and rotating the bottle by hand for at least one minute. A bottle containing unused material should be recapped immediately and stored at room temperature away from light.

To use the uncertainty estimates given in this certificate, divide the expanded uncertainty by $k = 2$ to obtain the combined standard uncertainty. The effective degrees of freedom of the combined standard uncertainty are ≥ 60.

PREPARATION AND ANALYSIS[(1)]

The material for SRM 59a was furnished by the Interlake Steel Corp. (Beverly, OH) in cake form. The cake material was processed by crushing, grinding and sieving at Strategic-UDY Processes, Inc. (Niagara Falls, NY). Additional mixing, blending, and sieving was done at NIST by G.E. Deardorff and H.L. Carter.

Homogeneity testing was performed at NIST using X-ray fluorescence spectrometry, gravimetric analysis, and neutron activation analysis by S.D. Rasberry, C.W. Gifford, R.K. Bell, and P.D. LaFleur. After additional processing, material variability was determined to be satisfactory for the certified constituents.

Each certified value is an unweighted mean of the results from the methods listed in Table 2. The uncertainty listed with each certified value is an expanded uncertainty about the mean, with coverage factor, $k = 2$, calculated following the ISO/JCGM Guide [10].

Analyses leading to the certification of this SRM were performed at NIST by J.R. Baldwin, R.K. Bell, E.R. Deardorff, C. Gifford, E.J. Maienthal, L. Moore, T.J. Murphy, T.C. Rains, S.D. Rasberry, K.R. Sappenfield, W.R. Shields, and S.A. Wicks, all formerly of the NIST Chemical Sciences Division. Analytical determinations were also performed by J.C. Cline and R.A. Pontello, Interlake, Inc. (Beverly, OH); L.F. Risi, Shieldalloy Corp. (Newfield, NJ); G. Porter, H.H. Hall and J.T. Waller, Union Carbide Corp. (Marietta, OH); and H.R. Grady, Foote Mineral Co. (Exton, PA).

NOTICE TO USERS

NIST strives to maintain the SRM inventory supply, but NIST cannot guarantee the continued or continuous supply of any specific SRM. Accordingly, NIST encourages the use of this SRM as a primary benchmark for the quality and accuracy of the user's in-house reference materials and working standards. As such, the SRM should be used to validate the more routinely used reference materials in a laboratory. Comparisons between the SRM and in-house reference materials or working measurement standards should take place at intervals appropriate to the conservation of the SRM and the stability of relevant in-house materials. For further guidance on how this approach can be implemented, contact NIST by email at srms@nist.gov.

[(1)] Certain commercial equipment, instruments or materials are identified in this certificate 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 materials or equipment identified are necessarily the best available for the purpose.

Table 2. Test Methods Employed in the Certification of SRM 59a

Element	Test Methods Used at NIST and Collaborating Laboratories
Aluminum	Polarography; Flame atomic absorption spectrometry (AAS); NaOH separation of Al, followed by titration with 1,2-diaminocyclohexanetetraacetic acid
Boron	Isotope dilution mass spectrometry (IDMS); Azure C photometric method; Spectrographic method
Calcium	Flame atomic absorption spectrometry (FAAS); Flame emission spectrometry (FES); EDTA titration; Ca precipitated as oxalate and titrated with standard $KMnO_4$ solution
Carbon	Combustion with gravimetric determination; Combustion with conductometric detection
Chromium	Flame atomic absorption spectrometry (FAAS); Cr oxidized with ammonium persulfate and potentiometric titration with ferrous ammonium sulfate; Diphenylcarbazide photometric method; Spectrometric method
Copper	Photometric method; Flame atomic absorption spectrometry (FAAS); Isotope dilution mass spectrometry (IDMS); Diethyldithiocarbamate photometric method; Neocuprine photometric method
Iron	Dissolved in nitric and hydrofluoric acids and fumed with H_2SO_4, Fe precipitated with $SnCl_2$ and titrated with $K_2Cr_2O_7$ standard solution; Fe reduced with $SnCl_2$ and titrated with $K_2Cr_2O_7$ standard solution; Fe titrated with $KMnO_4$ standard solution;
Manganese	Persulfate-arsenite titration method; Sodium pyrophosphate method; KIO_4 photometric method
Nickel	Flame atomic absorption spectrometry (FAAS)
Phosphorus	Photometric method
Silicon	Double dehydration with $HClO_4$; Fusion with Na_2O_2 and double dehydration with HCl

ADDITIONAL CONSTITUENTS: Noncertified values are provided for the following additional constituents in SRM 59a.

Information Mass Fraction Value: An information value for sulfur mass fraction, measured using combustion with titration and combustion with infrared detection, is reported in Table 3. An information value is a value that may be of interest to the SRM user, but insufficient information is available to assess the uncertainty associated with the value. Information values cannot be used to establish metrological traceability.

Table 3. Information Mass Fraction Value for SRM 59a

Constituent	Mass Fraction (%)
Sulfur (S)	0.002

REFERENCES

[1] Thompson, A.; Taylor, B.N.; *Guide for the Use of the International System of Units (SI)*; NIST Special Publication 811; U.S. Government Printing Office: Washington, DC (2008); available at https://www.nist.gov/pml/pubs/sp811/index.cfm (accessed Jun 2019).

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[3] Adcock, R.J., *A Problem in Least Squares.* The Analyst, Vol. 5, pp. 53–54 (1878).

[4] Deming, W.E., *Statistical Adjustment of Data*, John Wiley & Sons, New York, NY (1943).

[5] Horwitz, W., *The Certainty of Uncertainty*. J. AOAC International, Vol. 86, pp. 109–111 (2003).

[6] Manuilova, E., Schuetzenmeister, A., and Model, F., *mcr: Method Comparison Regression* (2014). https://CRAN.R-project.org/package=mcr; R package version 1.2.1 (accessed Feb 2019).

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[8] Thompson, M., *The Amazing Horwitz Function*, AMC Technical Brief 17, Royal Soc. of Chemistry (2004).

[9] Thompson, M., *Limitations of the Application of the Horwitz Equation: A Rebuttal;* Trends in Analytical Chemistry, Vol. 26, pp. 659–661 (2007).

[10] JCGM 100:2008; *Evaluation of Measurement Data - Guide to the Expression of Uncertainty in Measurement*; (ISO GUM 1995 with Minor Corrections), Joint Committee for Guides in Metrology (JCGM) (2008); available at https://www.bipm.org/utils/common/documents/jcgm/JCGM_100_2008_E.pdf (accessed Jun 2019); see also Taylor, B.N.; Kuyatt, C.E.; *Guidelines for Evaluating and Expressing the Uncertainty of NIST Measurement Results*; NIST Technical Note 1297, U.S. Government Printing Office: Washington, DC (1994); available at https://www.nist.gov/pml/pubs/index.cfm (accessed Jun 2019).

Certificate Revision History: 11 June 2019 (Analyst name correction; editorial changes); 05 March 2019 (Correction to revision history; editorial changes); 24 August 2018 (Revised values and uncertainties for all certified values, reassignment of the sulfur value as an information value; title update; editorial changes); 6 November 1969 (Original certificate date); 27 August 1969 (Provisional 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 at https://www.nist.gov/srm.