National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material® 68c

Standard Ferromanganese
(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 ferromanganese and materials of similar matrix. It can be used to validate value assignment of in-house reference materials. A unit of SRM 68c consists of one bottle containing approximately 100 g of powder with particle sizes in the range of 0.15 mm to 0.25 mm.

Certified Mass Fraction Values: Certified values for constituents of SRM 68c are reported in Table 1 as mass fractions of the elements in a ferromanganese 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 68c

Constituent	Mass Fraction (%)	Expanded Uncertainty (%)
Arsenic (As)	0.0212	0.0021
Carbon (C)	6.721	0.031
Chromium (Cr)	0.0744	0.0069
Iron (Fe)	12.30	0.16
Manganese (Mn)	80.04	0.11
Phosphorus (P)	0.192	0.010
Silicon (Si)	0.2250	0.0063

Expiration of Certification: The certification of **SRM 68c** 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.

Coordination of technical measurements for certification was performed by 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.

Carlos A. Gonzalez, Chief
Chemical Sciences Division

Gaithersburg, MD 20899
Certificate Issue Date: 05 March 2019
Certificate Revision History on Last Page

Steven J. Choquette, Director
Office of Reference Materials

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

INSTRUCTIONS FOR HANDLING, STORAGE, AND USE

Ferromanganese 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 68c was provided and processed by Union Carbide Corporation, Metals Division (Marietta, OH), through the courtesy of H.H. Hall. A test portion was subjected to a sieve fraction study at NIST. This was followed by sieving the material, primarily to remove fines, and by subsequent blending.

Homogeneity testing was performed at NIST by direct neutron activation analysis to measure Mn in disks made from 250 mg to 300 mg of powder on polyester tape, by combustion analysis for C, and by titrimetry for Fe. The material variability was determined to be within the imprecision of each method.

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 [3−10].

Analyses leading to the certification of this SRM were performed at NIST by E.R. Deardorff, B.I. Diamondstone and R.F. Fleming formerly of the NIST Chemical Sciences Division. Analytical determinations were also performed by A.I. Fulton and C.W. Hartig, Allegheny Ludlum Steel Corp. (Brackenridge, PA); J.E. Joyce, Inland Steel (East Chicago, IN); J.C. Cline and R.A. Pontella, Interlake, Inc. (Beverly, OH); L.F. Risi, Shieldalloy Corp. (Newfield, NJ); J.E. Cumbo, E.W. Linton and P.J. Butry, SKW Alloys, Inc. (Niagara Falls, NY); and H.H. Hall, Union Carbide Corp. (Marietta, OH).

Table 2. Test Methods Employed in the Certification of SRM 68c

Element	Test Methods Used at NIST and Collaborating Laboratories
Arsenic	Photometry; Distillation-H_2S-As_2S_3; X-ray fluorescence (XRF) spectrometry
Carbon	Combustion with infrared detection
Chromium	Atomic absorption spectrometry (AAS); Na_2O_2 oxidation – $FeSO_4$-$KMnO_4$ titration; X-ray fluorescence (XRF) spectrometry
Iron	Titrimetry
Manganese	Potentiometric titration with $KMnO_4$ in neutral pyrophosphate solution; Bismuthate-$FeSO_4$-$KMnO_4$ titration; Manganese oxidized with $HClO_4$-H_3PO_4 acids – $FeSO_4$-$KMnO_4$ titration; DC plasma emission spectrometry
Phosphorus	Alkalimetry; Molybdovanadophosphate photometry; Weighed as $(NH_4)_3PMo_{12}O_{40}$
Silicon	$HClO_4$ dehydration; K_2SiF_6 precipitation followed by NaOH titration; H_2SO_4 dehydration

ADDITIONAL CONSTITUENTS: Noncertified values are provided for the following additional constituents in SRM 68c.

Information Mass Fraction Values: Information values for constituents in SRM 68c are reported as mass fractions 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 [2]. Information values cannot be used to establish

[(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.

metrological traceability. The information values reported in Table 3 were determined using a single test method at NIST.

Table 3. Information Mass Fraction Values for SRM 68c

Constituent	Mass Fraction (%)
Cobalt (Co)	0.18
Nickel (Ni)	0.12
Oxygen (O)	0.11
Sulfur (S)	0.008

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.

REFERENCES

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Certificate Revision History: 05 March 2019 (Correction to revision history; editorial changes); 24 August 2018 (Revised certified values for As, C, Cr, P, and Si; revised uncertainties for all certified values, reassignment of the sulfur value as an information value; addition of information values for Co, Ni and O; title update; editorial changes); 15 August 1979 (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 at https://www.nist.gov/srm.