National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material® 64c

Ferrochromium
High Carbon
(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 ferrochromium and materials of similar matrix. It can be used to validate value assignment of in-house reference materials. A unit of SRM 64c consists of one bottle containing approximately 100 g of fine powder less than 150 µm (100 mesh).

Certified Mass Fraction Values: Certified values for constituents of SRM 64c are reported in Table 1 as mass fractions of the elements in a ferrochromium 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 64c

Constituent	Mass Fraction (%)	Expanded Uncertainty (%)
Carbon (C)	4.698	0.040
Chromium (Cr)	68.00	0.20
Cobalt (Co)	0.0515	0.0037
Copper (Cu)	0.0053	0.0015
Iron (Fe)	24.99	0.12
Manganese (Mn)	0.1624	0.0090
Nickel (Ni)	0.429	0.019
Nitrogen (N)	0.0449	0.0037
Phosphorus (P)	0.0193	0.0020
Silicon (Si)	1.216	0.027
Sulfur (S)	0.0673	0.0034
Titanium (Ti)	0.0179	0.0061
Vanadium (V)	0.1528	0.0055

Expiration of Certification: The certification of **SRM 64c** 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.

Carlos A. Gonzalez, Chief
Chemical Sciences Division

Gaithersburg, MD 20899

Steven J. Choquette, Director
Office of Reference Materials

Certificate Issue Date: 05 March 2019
Certificate Revision History on Last Page

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.

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

INSTRUCTIONS FOR HANDLING, STORAGE, AND USE

Ferrochromium 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 500 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. The user should obtain a representative sample of the coarse and fine particles to ensure accurate measurements. 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 64c was provided by Airco Alloys (Niagara Falls, NY) through the courtesy of J.E. Cumbo. Crushing, grinding and sieving was performed by Union Carbide Corp. (Marietta, OH) through the courtesy of G. Porter. At NIST, the material was sieved, blended and bottled.

Homogeneity testing of samples from the final lot was performed by J.E. Cumbo, Airco Alloys (Niagara Falls, NY) and J.C. Cline, Interlake, Inc. (Beverly, OH).

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 S.A. Wicks (retired) of the NIST Chemical Sciences Division. Analytical determinations were also performed by J.E. Cumbo, Airco Alloys (Niagara Falls, NY); M. Dannis, Armco Steel Corp. (Middletown, OH); F.A. Blair, J.R. Carson, J.M. Arrit, D.E. Howells, F.E. Lowry, E.L. Montgomery, A.H. Roberts, E.B. Sharp, L.J. Stiles, and W.L. Stickler, Huntington Alloys, Inc. (Huntington, WV); J.C. Cline, Interlake, Inc. (Beverly, OH); L.F. Risi, Shieldalloy Corp. (Newfield, NJ); and G. Porter, H.H. Hall, Union Carbide Corp. (Marietta, OH).

(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 64c

Element	Test Methods Used at NIST and Collaborating Laboratories
Carbon	Combustion with chromatographic detection
Chromium	Na_2O_2 fusion – $FeSO_4$-$KMnO_4$ titration; Na_2O_2 fusion – $(NH_4)S_2O_8$ oxidation – potentiometric titration with standard $Fe(NH_4)_2(SO_4)_2$
Cobalt	Atomic Absorption spectrometry (AAS); Nitroso R photometric method; Spectrographic method
Copper	Atomic absorption spectrometry (AAS); Diethyldithiocarbamate photometric method
Iron	Na2O2 fusion – NH_4OH precipitation – $SnCl_2$-$KMnO_4$ titration; Acid dissolution – NH4OH precipitation – ceric sulfate potentiometric titration
Manganese	Peroxydisulfate – arsenite titration; KIO_4 photometric method; Spectrographic method; Atomic absorption spectrometry (AAS)
Nickel	Atomic absorption spectrometry (AAS); Weighed as nickel dimethylglyoxime; Spectrographic method
Nitrogen	Distillation and titration; Inert gas fusion with thermal conductivity detection (TCD)
Phosphorus	Photometric method; Gravimetric method; Ammonium phosphovanadate photometric method
Silicon	$HClO_4$ dehydration; Double dehydration; K_2SiF_6 precipitation followed by NaOH titration
Sulfur	Combustion with iodate titration; Combustion with chromatographic detection
Titanium	Photometric method; H_2O_2 photometric method; Spectrographic method; Anion exchange separation followed by H_2O_2 photometric method
Vanadium	Na_2O_2 fusion – $(NH_4)S_2O_8$ oxidation – $KMnO_4$ titration; H_2O_2 photometric method; Spectrographic method

ADDITIONAL CONSTITUENTS: Noncertified values are provided for the following additional constituents in SRM 64c.

Information Mass Fraction Values: Information values for constituents in SRM 64c 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 metrological traceability.

Table 3. Information Mass Fraction Values for SRM 64c

Constituent	Mass Fraction (%)
Aluminum (Al)	0.006
Arsenic (As)	0.003
Molybdenum (Mo)	< 0.001
Oxygen (O_2)	0.12
Tin (Sn)	< 0.0005

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

[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 Mar 2019).

[2] May, W.; Parris, R.; Beck II, C.; Fassett, J.; Greenberg, R.; Guenther, F.; Kramer, G.; Wise, S.; Gills, T.; Colbert, J.; Gettings, R.; MacDonald, B.; *Definition of Terms and Modes Used at NIST for Value-Assignment of Reference Materials for Chemical Measurements*; NIST Special Publication 260-136 U.S. Government Printing Office: Washington, DC (2000); available at https://www.nist.gov/srm/upload/SP260-136.PDF (accessed Mar 2019).

[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).

[7] R Core Team, *R: A Language and Environment for Statistical Computing*. R Foundation for Statistical Computing, Vienna, Austria (2015). https://www.r-project.org (accessed Feb 2019).

[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 Mar 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/nist-technical-note-1297 (accessed Mar 2019).

Certificate Revision History: 05 March 2019 (Correction to revision history; editorial changes); 24 August 2018 (Revised values and uncertainties for all certified values, title update; editorial changes); 20 February 1992 (Editorial revisions); 24 August 1977 (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.