National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material® 131h

Refined Cast Iron

This Standard Reference Material (SRM) is intended primarily for use in validation of chemical and instrumental methods of analysis. It can be used to validate value assignment of in-house reference materials and, if necessary, to calibrate carbon/sulfur analyzers. A unit of SRM 131h consists of a bottle containing 100 g of chips.

Certified Mass Fraction Value: The certified mass fraction value for sulfur in SRM 131h is listed in Table 1 [1]. Value assignment categories are based on the definitions of terms and modes used at NIST for certification of chemical reference materials [2]. 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.

Reference Mass Fraction Value: The reference mass fraction value for carbon in SRM 131h is listed in Table 2. A reference value is a non-certified value that is the present best estimate of the true value; however, the value does not meet the NIST criteria for certification [2] and is provided with an associated uncertainty that may reflect only measurement precision, may not include all sources of uncertainty, or may reflect a lack of sufficient agreement among multiple analytical methods.

Expiration of Certification: The certification of **SRM 131h** is valid indefinitely, within the measurement uncertainties specified, provided the SRM is handled and stored in accordance with the instructions given in this certificate (see "Instructions for Use"). Accordingly, periodic recalibration or 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, NIST will notify the purchaser. Registration (see attached sheet or register online) will facilitate notification.

Coordination of technical measurements for the certification of this SRM was performed by J.R. Sieber of the NIST Chemical Sciences Division.

Measurements for value assignment of SRM 131h were performed by J.R. Sieber of the NIST Chemical Sciences Division. Additional measurements were performed by collaborating laboratories: E. Hagen, Alcoa Howmet Research Center (Whitehall, MI); G. Mann, Anderson Laboratories, Inc. (Greendale, WI); T.A. Herdlein and K.L. Placta, ATI Specialty Materials (Lockport, NY); M. VanDyke, Cannon-Muskegon Corp. (North Shores, MI); and J. Gast, LECO Corp. (Saint Joseph, MI).

Statistical consultation for this SRM was provided by A.L. Pintar 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: 22 January 2016

Steven J. Choquette, Acting Director
Office of Reference Materials

INSTRUCTIONS FOR USE

To relate analytical determinations to the values in this Certificate of Analysis, a minimum sample quantity of 200 mg is recommended. The chips may be weighed directly for analysis. Store the material in its tightly-capped, original container in a cool, dry location. This material is not intended for frequent use as a routine quality assurance material.

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.

PREPARATION AND ANALYSIS[(1)]

The material for the preparation of this SRM was provided by Huntington Alloys, Inc. (Huntington, WV). The cast iron was refined to reduce levels of carbon and sulfur, and cast in billet form. The material was chipped, blended and bottled at the NIST facilities (Gaithersburg, MD).

Homogeneity testing was performed at the collaborating laboratories. The homogeneity of carbon and sulfur was found to be satisfactory at sample masses typically used for testing of produced iron and steel. At NIST, testing of sulfur was performed at nanogram mass levels, and the material was found to be sufficiently homogeneous. Test methods used in the development of this SRM were the following.

1. Collaborating laboratories used ASTM International E1019 Standard Test Methods for Determination of Carbon, Sulfur, Nitrogen, and Oxygen in Steel, Iron, Nickel, and Cobalt Alloys by Various Combustion and Fusion Techniques [3]. For carbon, the procedure *Carbon, Total, by the Combustion–Instrumental Measurement Test Method* was used. For sulfur, the procedure *Sulfur by the Combustion-Infrared Absorption Test Method (Calibration with Metal Reference Materials)* was used.

2. At NIST, energy-dispersive X-ray fluorescence (XRF) spectrometry was performed using a microXRF spectrometer to measure small portions of individual chips of iron and steel. Calibration was done using selected chip form Standard Reference Materials.

Certified Mass Fraction Value: The assigned value for sulfur and 95 % coverage interval are given in Table 1. The measurand is the mass fraction of total sulfur in cast iron. The certified value is an estimate of the true average value of the measurand over all bottles of SRM 131h, and the estimate comes from fitting a statistical model to the results of measurements made directly on the SRM 131h material using two test methods. The Bayesian paradigm was used for statistical inference [4]. The expanded uncertainty is an interval calculated in a manner consistent with the ISO/JCGM Guides [5,6], and it expresses contributions from all recognized sources of uncertainty, including differences between analytical methods, dispersion of values resulting from sample preparation and replicated measurement, preparation and measurement of calibrants, analytical calibration functions, and balance calibration. The certified mass fraction value in Table 1 is metrologically traceable to the derived SI unit for mass fraction expressed as milligrams per kilogram.

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

Table 1. Certified Mass Fraction Value for SRM 131h Refined Cast Iron

Constituent	Mass Fraction (mg/kg)	95 % Coverage Interval (mg/kg)
Sulfur (S)	7.4	6.0 to 9.1

Reference Mass Fraction Value: The assigned value for carbon and 95 % coverage interval are given in Table 2. The measurand is the mass fraction of total carbon in cast iron as determined using the test method in ASTM E1019-11. The reference value is an estimate of the true average value of the measurand over all bottles of SRM 131h, and the estimate comes from fitting a statistical model to the results of measurements made directly on the SRM 131h material using the test method in ASTM E1019. The Bayesian paradigm was used for statistical inference [4]. The expanded uncertainty is an interval calculated in a manner consistent with the ISO/JCGM Guides [5,6], and it expresses contributions from all recognized sources of uncertainty, including differences between analytical methods, dispersion of values resulting from sample preparation and replicated measurement, preparation and measurement of calibrants, analytical calibration functions, and balance calibration. The reference mass fraction value in Table 2 is metrologically traceable to the derived SI unit for mass fraction expressed as milligrams per kilogram.

Table 2. Reference Mass Fraction Value for SRM 131h Refined Cast Iron

Constituent	Mass Fraction (mg/kg)	95 % Coverage Interval (mg/kg)
Carbon (C)	7.8	5.4 to 11

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 www.nist.gov/pml/pubs/index.cfm/ (accessed Jan 2016).

[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.; *Definitions of Terms and Modes Used at NIST for Value Assignment of Reference Materials for Chemical Measurements*; NIST Special Publication 260-136 (2000); available at http://www.nist.gov/srm/upload/SP260-136.PDF (accessed Jan 2016).

[3] ASTM E1019-11; *Standard Test Methods for Determination of Carbon, Sulfur, Nitrogen, and Oxygen in Steel, Iron, Nickel, and Cobalt Alloys by Various Combustion and Fusion Techniques*; ASTM International, West Conshohocken, PA (2009); available at www.astm.org (accessed Jan 2016).

[4] Gelman, A.; Carlin, J.B.; Stern, H.S.; Dunson, D.B.; Vehtari, A.; Rubin, D.B.; *Bayesian Data Analysis*; 3rd ed., CRC Press (2014).

[5] JCGM 100:2008; *Evaluation of Measurement Data — Guide to the Expression of Uncertainty in Measurement*; (GUM 1995 with Minor Corrections), Joint Committee for Guides in Metrology (JCGM) (2008); available at http://www.bipm.org/utils/common/documents/jcgm/JCGM_100_2008_E.pdf (accessed Jan 2016); 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 http://www.nist.gov/pml/pubs/index.cfm (accessed Jan 2016).

[6] JCGM 101:2008; *Evaluation of Measurement Data – Supplement 1 to the Guide to the Expression of Uncertainty in Measurement – Propagation of Distributions Using a Monte Carlo Method*; JCGM (2008); available at http://www.bipm.org/utils/common/documents/jcgm/JCGM_101_2008_E.pdf (accessed Jan 2016).

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 http://www.nist.gov/srm..