National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material® 361

AISI 4340 Steel
(chip form)

This Standard Reference Material (SRM) is intended primarily for use in chemical methods of analysis. A unit of SRM 361 consists of a bottle containing approximately 150 g of chips sized between 0.5 mm and 1.18 mm sieve openings (35 mesh and 16 mesh).

Certified Mass Fraction Values: Certified mass fraction values for elements in SRM 361 are 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.

Information Values: Information mass fractions values for elements in SRM 361 are listed in Table 2 [1]. An information value is considered to be a value that will be of interest to the SRM user, but insufficient information is available to assess the uncertainty associated with the value. They are intended to provide additional information on the matrix. Information values cannot be used to establish metrological traceability.

Expiration of Certification: The certification of **SRM 361** is valid indefinitely, within the uncertainty 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.

Overall direction and coordination of the original technical measurements leading to certification were performed by O. Menis, B.F. Scribner, and J.I. Shultz, formerly of the NIST Office or Reference Materials. Coordination of boron measurements for certification was performed by R.R. Greenberg of the NIST Chemical Sciences Division.

Measurements for value assignment of SRM 361 were performed by R. Alvarez, D.A. Becker, E.L. Garner, T.E. Gills, R.M. Lindstrom, E.J. Maienthal, C.W. Mueller, P.J. Paulsen, K.M. Sappenfield, B.A. Thompson, J.L. Weber, Jr., S.A. Wicks, and J. Wing formerly of the NIST Chemical Sciences Division. Additional measurements were performed by collaborating laboratories: R.H. Rouse, Bethlehem Steel Corp., Sparrows Point Plant, MD; E.J. Cramer, Carpenter Technology Corp., Reading, PA; R.G. Cover, The Timken Roller Bearing Co., Canton, OH; L.M. Melnick, United States Steel Corp., Monroeville, PA; and E.H. Shipley, Gary Steel Works, Gary, IN.

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

Steven J. Choquette, Director
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Gaithersburg, MD 20899
Certificate Issue Date: 22 September 2016
Certificate Revision History on Last Page

INSTRUCTIONS FOR USE

To relate analytical determinations to the certified values on this Certificate of Analysis, a minimum sample quantity of 200 mg is recommended. Specimens may be used directly from the bottle without pre-treatment. The material should be stored in its original container in a cool, dry location.

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 this reference material was vacuum melted and cast by the Carpenter Technology Corp., Reading, PA to provide material of the highest possible homogeneity. The work was made possible by a grant from the American Iron and Steel Institute (AISI). Following acceptance of the material, selected portions of the ingots were extensively tested for homogeneity by D.M. Bouchette, S.D. Rasberry, and J.L. Weber, Jr., formerly of the NIST Chemical Sciences Division. Certification analyses were made on composite samples. However, for certain elements and based on previous experience, only one composite sample was analyzed with the results applied to the other forms of the material.

Certified Mass Fraction Values: The measurand is the total elemental content for each element listed in Table 1. Metrological traceability is to the derived SI unit for mass fraction, expressed as a percent or milligrams per kilogram. The uncertainties for all elements, with the exception of boron, are given as combined standard uncertainty estimates, u_c, where u_c is intended to represent, at the level of one standard deviation, the combined effect of uncertainty components associated with the measurements and with element inhomogeneity, and reflect the guidance given in NBS Monograph 148 [3]. The uncertainty for boron is assessed according to the ISO/JCGM Guide [4]. The certified values for boron and sulfur were determined by thermal prompt gamma activation analysis and isotope dilution spark source mass spectrometry at NIST, respectively. The boron value was confirmed using isotope dilution inductively coupled plasma mass spectrometry and inductively coupled plasma optical emissions spectrometry by Coedo et al. [5], and the sulfur value was confirmed using isotope dilution mass spectrometry data provided by the Japan Atomic Energy Research Institute (JAERI). The expanded uncertainty for boron is calculated as $U = ku_c$, where u_c is intended to represent, at the level of one standard deviation, the combined effect of uncertainty components associated with the measurements and with element inhomogeneity. The coverage factor, $k = 2$, is determined from the Student's t-distribution with 5.9 degrees of freedom and corresponds to an approximate 95 % confidence interval. The other elements were measured at NIST and the cooperating laboratories using a variety of chemical methods. The certified values and uncertainties for these elements are the present best estimates of the true values based on the results of the cooperative analytical program.

(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 Institute 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 Values for SRM 361

Element	Mass Fraction (%)			Element	Mass Fraction (%)		
Aluminum (total)	0.021	±	0.005	Neodymium	0.00075	±	0.00005
Antimony	0.0042	±	0.0001	Nickel	2.00	±	0.01
Arsenic	0.017	±	0.001	Niobium	0.022	±	0.001
Calcium	0.00010	±	0.00005	Phosphorus	0.014	±	0.001
Carbon	0.383	±	0.001	Silicon	0.222	±	0.001
Cerium	0.0040	±	0.0001	Silver	0.0004	±	0.0001
Chromium	0.694	±	0.005	Sulfur	0.0143	±	0.0003
Cobalt	0.032	±	0.001	Tantalum	0.020	±	0.001
Copper	0.042	±	0.001	Tin	0.010	±	0.001
Lead	0.000025	±	0.000005	Titanium	0.020	±	0.001
Magnesium	0.00026	±	0.00005	Tungsten	0.017	±	0.001
Manganese	0.66	±	0.01	Vanadium	0.011	±	0.001
Molybdenum	0.19	±	0.01	Zirconium	0.009	±	0.001
Boron	4.78 mg/kg	±	0.15 mg/kg				

Information Mass Fraction Values: The information value for each analyte is an estimate obtained from one or more NIST or collaborator test methods. No uncertainty is provided because there is insufficient information available for its assessment.

Table 2. Information Mass Fraction Values for SRM 361

Element	Mass Fraction (%)	Element	Mass Fraction (%)
Bismuth	0.0004	Oxygen	0.0009
Gold	< 0.00005	Praseodymium	0.0003
Hafnium	0.0002	Selenium	0.004
Hydrogen	< 0.0005	Strontium[a]	< 0.0005
Iron (by difference)	95.6	Tellurium	0.0006
Lanthanum	0.001	Zinc	0.0001
Nitrogen	0.0037		

[a] Element "not detected."

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 Sep 2016).

[2] May, W.; Parris, R.; Beck, 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; U.S. Government Printing Office: Washington, DC (2000); available at www.nist.gov/srm/publications.cfm (accessed Sep 2016).

[3] Cali, J.P., et al.; *The Role of Standard Reference Materials in Measurement Systems*; NBS Monograph 148, p. 21, (1975).

[4] 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 (2008); available at www.bipm.org/utils/common/documents/jcgm/JCGM_100_2008_E.pdf (accessed Sep 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 www.nist.gov/pml/pubs/tn1297/index.cfm (accessed Sep 2016).

[5] Coedo, A.G.; Dorado, T.; Fernandez, B.J.; Alguacil, F.J.; *Isotope Dilution Analysis for Flow Injection ICPMS Determination of Microgram per Gram Levels of Boron in Iron and Steel after Matrix Removal*; Analytical Chemistry, Vol. 68, pp. 991–996, (1996).

Certificate Revision History: 22 September 2016 (Change of expiration date; editorial changes); 29 May 2001 (This revision reflects the addition of a new boron value and editorial changes); 20 May 1996 (This revision reflects the removal of the boron value and editorial changes); 24 February 1981 (This revision reflects a change in the sulfur value); 05 June 1979 (This revision reflects changes in the boron, cobalt, and sulfur values); 08 January 1976 (This revision reflects the addition of nine certified elements); 26 July 1970 (Originally issued as a provisional certificate).

Users of this SRM should ensure that the Certificate 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.