Certificate of Analysis

Standard Reference Material® 2165

Low Alloy Steel
(chip form)

This Standard Reference Material (SRM) is low alloy steel in the form of chips sized to pass through sieve openings between 0.50 mm and 1.18 mm (35 mesh to 16 mesh). It is intended primarily for use in evaluation of chemical and instrumental methods of analysis of steel and materials of similar matrix. It should not be used for calibration. A unit of SRM 2165 consists of one bottle containing approximately 150 g of chips.

Certified Mass Fraction Values: Certified values for 16 constituents of SRM 2165 are reported in Table 1 as mass fractions [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 investigated or taken into account [2]. A certified value is the present best estimate of the true value based on the results of analyses performed at NIST and collaborating laboratories using the test methods shown in Table 4.

Reference Mass Fraction Values: Reference values for two constituents are reported in Table 2. Reference values are non-certified values that are the present best estimates of the true values. However, the values do not meet the NIST criteria for certification and are provided with associated uncertainties that may not include all sources of uncertainty [2].

Information Mass Fraction Values: Information values for seven constituents are reported in Table 3. 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.

Expiration of Certification: The certification of **SRM 2165** 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 material 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) will facilitate notification.

Coordination of the original certification of SRM 2165 was performed by J.I. Schultz, ASTM/NIST Research Associate, and W.P. Reed of NIST. Coordination of the reevaluation of boron, carbon, and sulfur contents was performed by J.R. Sieber of the NIST Analytical Chemistry Division.

Analyses in the original characterization were performed by J.A. Norris, D.E. Brown, and R.C. Gauer of what is now the NIST Chemical Sciences Division.

Analyses for the update were performed by W.R. Kelly, J.L. Mann, R.L. Paul, and R.D. Vocke of the NIST Chemical Sciences Division.

Statistical consultation for this SRM was provided by W.F. Guthrie of the NIST Statistical Engineering Division.

Carlos A. Gonzalez, Chief
Chemical Sciences Division

Gaithersburg, MD 20899

Steven J. Choquette, Director
Office of Reference Materials

Certificate Issue Date: 08 February 2018
Certificate Revision History on Last Page

Analyses for certification were also performed in the following laboratories: Allegheny Ludlum Steel Corp. (Brackenridge, PA) by R.M. Crain, G.L. Bergstrom, and C.C. Gabrielli; Analytical Associates, Inc. (Detroit, MI) by C.K. Deak; Armco Research & Technology (Middletown, OH) by C.C. Borland, D.E. Gillum, and H.P. Vail; General Motors Research Laboratories (Warren, MI) by M.P. Balogh, R.L. Passeno, W.S. Antos, and N.M. Potter; Institut de Recherches de la Sidérurgie Française (Maizieres-Les-Metz, France) by D. Ravaine; Ledoux & Co. (Teaneck, NJ) by S. Kallmann and C.L. Maul.

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

INSTRUCTIONS FOR USE

To relate analytical determinations to certified values in 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 tightly sealed, original bottle in a cool, dry location.

Preparation and Analysis[(1)]: The material for SRM 2165 was vacuum induction melted at the Carpenter Technology Corp., Reading, PA and supplied in the form of rods. The material was cut and packaged at NIST in the NIST Measurement Services Division. Homogeneity testing was performed at NIST and at Lukens Steel Co., Coatesville, PA using spark source optical emission spectrometry.

Certified Value Assignment: Unless otherwise indicated, the certified values are the equally weighted means of results obtained by NIST and the collaborating laboratories. The uncertainty is expressed as the combined uncertainty, u_c, based on a combination of replicate measurement variability and an estimation of the combined effects of method variability, possible systematic errors among methods, and material variability. The estimated effective degrees of freedom for u_c is three. The measurands are the certified values listed in Table 1. The certified values are metrologically traceable to the SI derived unit of mass fraction (expressed as percent).

The certified value for sulfur was obtained by regression of results measured by NIST using isotope dilution thermal ionization mass spectrometry. The expanded uncertainty is calculated as $U = ku_c$ where u_c is the combined uncertainty at the level of one standard deviation, and the coverage factor, $k = 2.37$, was determined from the Student's *t*-distribution corresponding to the associated degrees of freedom and 95 % confidence level [3]. The measurand is the total concentration of Sulfur, and the certified value is metrologically traceable to the SI unit for mass, expressed as mass fraction.

Table 1. Certified Mass Fraction Values for SRM 2165

Constituent	Mass Fraction (%)		
Antimony (Sb)	0.001 0	±	0.000 5
Arsenic (As)	0.001 0	±	0.000 5
Chromium (Cr)	0.050	±	0.002
Cobalt (Co)	0.001 2	±	0.000 2
Copper (Cu)	0.001 3	±	0.000 2
Lead (Pb)	0.000 3	±	0.000 1
Manganese (Mn)	0.144	±	0.003
Molybdenum (Mo)	0.005 5	±	0.000 5
Nickel (Ni)	0.155	±	0.002
Niobium (Nb)	0.000 4	±	0.000 1
Phosphorus (P)	0.005 2	±	0.000 2
Silver (Ag)	0.000 2	±	0.000 1
Sulfur (S)	0.003 643	±	0.000 031
Tin (Sn)	0.002	±	0.001
Titanium (Ti)	0.005 1	±	0.000 2
Vanadium (V)	0.004 0	±	0.000 2

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

Reference Value Assignment: The measurands are the concentrations of the constituents listed in Table 2 as determined by the methods indicated in Table 4. Metrological traceability is to the SI derived unit for mass fraction (expressed as percent). The value for boron is the mean of results obtained by NIST using prompt gamma-ray activation analysis. The expanded uncertainty is calculated as $U = ku_c$ where u_c is the combined uncertainty at the level of one standard deviation, and the coverage factor, $k = 1.99$, was determined from the Student's t-distribution corresponding to the associated degrees of freedom and 95 % confidence level [3]. The value for carbon is based on results obtained by multiple laboratories using combustion methods. To best account for the different sources of variation in the interlaboratory data for the carbon mass fraction, a Bayesian hierarchical model with non-informative prior distributions was used to establish the mean mass fraction and its expanded uncertainty, U, which is given as a symmetric 95 % probability interval [4–6]. The expanded uncertainty can be expressed as $U = ku_c$, where $u_c = 5.58$ mg/kg is the combined standard uncertainty, and the coverage factor, $k = 2.07$, is determined from the Student's t-distribution corresponding to 22.9 degrees of freedom. The results of the Bayesian analysis can be interpreted in essentially the same way as results from the ISO approach using the values given above and in the body of Table 1. Alternatively, a discrete approximation of the posterior distribution for the certified carbon mass fraction is provided in reference 7 for subsequent Bayesian uncertainty calculations.

Table 2. Reference Mass Fraction Values for SRM 2165

Constituent	Mass Fraction (mg/kg)
Boron (B)	9.44 ± 0.17
Carbon (C)	63 ± 12

Information Value Assignment: The values reported are estimates based on technical evaluation of the results reported from one or more test methods performed by the collaborating laboratories. Information values cannot be used to establish metrological traceability.

Table 3. Information Mass Fraction Values for SRM 2165

Constituent	Mass Fraction (mg/kg)
Aluminum (Al)	60
Bismuth (Bi)	<1
Magnesium (Mg)	<1
Selenium (Se)	35
Silicon (Si)	40
Tantalum (Ta)	40
Tellurium (Te)	30

Table 4. Test Methods for NIST Analysis of SRM 2165

Combustion with infrared or thermal conductivity detection:	C, S
Direct current plasma optical emission spectrometry:	Ag, Al, Co, Cr, Cu, Mg, Mn, Mo, Nb, Ni, P, Si, Ti, V, Zr
Flame atomic absorption spectrophotometry:	Cr, Cu, Mn, Ni, Sn, Ti, V
Inductively coupled plasma optical emission spectrometry:	Ag, As, Co, Cr, Cu, Mn, Mo, Nb, Ni, P, Pb, Sb, Si, Sn, Ti, V
Inert gas fusion with infrared detection:	C, S
Isotope dilution thermal ionization mass spectrometry:	S
Photometric methods:	Mn, P
Prompt gamma-ray activation analysis:	B
Zeeman atomic absorption spectrophotometry:	Ag, As, Bi, Pb, Sb, S

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/index.cfm/ (accessed Feb 2018).

[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 https://www.nist.gov/srm/publications.cfm (accessed Feb 2018).

[3] 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 http://www.bipm.org/utils/common/documents/jcgm/JCGM_100_2008_E.pdf (accessed Feb 2018); 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 Feb 2018).

[4] Lee, P.M.; *Bayesian Statistics: An Introduction*, 2nd ed.; Arnold: London, p. 344 (1997).

[5] Gelman, A.; Carlin, J.B.; Stern, H.S.; Rubin, D.B.; *Bayesian Data Analysis*; Chapman and Hall: London, p. 526 (1995).

[6] Lunn, D.J.; Thomas, A.; Best, N.; Spiegelhalter, D.; WinBUGS – A Bayesian Modelling Framework: Concepts, Structure, and Extensibility; *Statistics and Computing*; Vol. 10, pp. 325–337 (2000).

[7] The file containing the approximation of the posterior distribution for carbon (See "Reference Value Assignment") can be obtained from the data link to the supplementary information on this material at: https://www-s.nist.gov/srmors/view_detail.cfm?srm=2165 (accessed Feb 2018).

Certificate Revision History: 08 February 2018 (Title change; editorial changes); 07 June 2011 (Update of values for boron, carbon, and sulfur based on new analytical determinations; editorial changes); 12 June 1989 (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 at: telephone (301) 975-2200; fax (301) 948-3730, email srminfo@nist.gov; or via the Internet at https:/www.nist.gov/srm.