National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material® 8k

Bessemer Steel (Simulated) 0.1 % Carbon
(chip form)

This Standard Reference Material (SRM) is intended primarily for use in validation of chemical and instrumental methods of analysis. A unit of SRM 8k consists of a bottle containing approximately 150 g of fine millings sized between 0.50 mm (No. 35 sieve) and 1.18 mm (No. 16 sieve).

Certified Values: Certified mass fraction values for constituents of SRM 8k are listed in Table 1 [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 uncertainties are expressed at a confidence level of approximately 95 % confidence.

Table 1. Certified Values for SRM 8k Bessemer Steel (Simulated) 0.1 % Carbon

Constituent	Mass Fraction (%)	Expanded Uncertainty (%)
Manganese (Mn)	0.5046	0.0083
Copper (Cu)	0.0200	0.0027
Chromium (Cr)	0.0467	0.0027
Vanadium (V)	0.0145	0.0011

Expiration of Certification: The certification of **SRM 8k** 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 Storage, Handling 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, NIST will notify the purchaser. Registration (see attached sheet or register online) will facilitate notification.

Coordination of technical measurements leading to certification was performed under the direction of J.R. Sieber of the NIST Chemical Sciences Division.

Statistical consultation for this SRM was provided by J.H. Yen 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
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Certificate Revision History on Last Page

Steven J. Choquette, Director
Office of Reference Materials

INSTRUCTIONS FOR STORAGE, HANDLING AND USE

To relate analytical determinations to the values on this Certificate of Analysis, a minimum sample quantity of 200 mg is recommended. The millings do not require preparation prior to weighing and dissolution. The material should be stored in its original container in a cool, dry location.

ADDITIONAL CONSTITUENTS: Noncertified values are provided for the following additional constituents in SRM 8k.

Reference Mass Fraction Values: Reference values for constituents of SRM 8k are listed in Table 2 . A reference value is a noncertified value that is the present best estimate of the true value based on available data; however, the value does not meet the NIST criteria for certification and is provided with associated uncertainties that may reflect only measurement repeatability, may not include all sources of uncertainty, or may reflect a lack of sufficient statistical agreement among multiple analytical methods [2]. The uncertainty listed with each value is an expanded uncertainty based on a 95 % confidence interval and is calculated according to the method in the JCGM and NIST Guides [3].

Table 2. Reference Values for SRM 8k Bessemer Steel (Simulated)

Constituent	Mass Fraction (%)	Expanded Uncertainty (%)
Carbon (C)	0.0806	0.0014
Phosphorus (P)	0.0956	0.0023
Sulfur (S)	0.0775	0.0050
Silicon (Si)	0.0576	0.0083
Nickel (Ni)	0.1174	0.0045
Molybdenum (Mo)	0.0397	0.0036

PREPARATION AND ANALYSIS[(1)]

SRM 8k was produced in cooperation with ASTM International Committee E01 Analytical Chemistry of Metals, Ores and Related Materials. The material for the preparation of this SRM was prepared by the Carpenter Technology Corporation, Reading, PA. This particular batch of steel was held in reserve after the certification of SRM 8j.

Certification of SRM 8k is based on a careful comparison of the reserve material to SRM 8j using a measurement procedure based on high-performance inductively coupled plasma optical emission spectrometry (ICP–OES) and the Bonferroni method of simultaneous statistical inference [4]. Except for Ni, all constituents were shown to be of the same composition in both materials. Due to a statistically significant difference between materials, Ni was determined using the ICP-OES measurements with SRM 8j as the calibrant.

For complete documentation, the details of planning, preparation, testing, and analysis for SRM 8j are included here.

The technical and support aspects involved in the original preparation and analysis of SRM 8j were coordinated by the NIST Office of Reference Materials by R.E. Michaelis.

Homogeneity testing was performed at NBS by optical emission spectrometric analysis by J.A. Norris; C/S analysis by B.I. Diamondstone; and selected chemical analyses by R.K. Bell (Assistant Research Associate, ASTM/NBS Research Associate Program). Test methods used in the development of SRM 8j are listed in Table 3

Cooperative analyses for certification were performed in the following laboratories: S.A. Wicks, R.K. Bell, and E.R. Deardorff, National Bureau of Standards, Institute for Materials Research, Analytical Chemistry Division; R.W. Bley, Inland Steel Company, East Chicago, IN; R.H. Rouse, Bethlehem Steel Corporation, Sparrows Point, MD; and F.P. Valente, U.S. Army Materials and Mechanics Research Center, Watertown, MA.

[(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 3. Analytical Methods Used to Determine the Element

Element	Methods
Carbon	Combustion – Infrared
Manganese	Persulfate Arsenite; $NaAsO_2$ Potentiometric Titration; KIO_4 Photometric Method
Phosphorus	Molybdate-Hydrazine Sulfate Photometric Method; Alkali-Molybdate; $Mg_2P_2O_7$ Gravimetric Method
Sulfur	Combustion – KIO_3 Titration
Silicon	Perchloric Acid Double Dehydration; Silicomolybdate Photometric Method
Copper	Diethyldithiocarbamate Photometric Method; Neocuproine Photometric Method
Nickel	Dimethylglyoxime Gravimetric Method; Dimethylglyoxime Spectrophotometric Method
Chromium	$NaHCO_3$ Hydrolysis – Peroxydisulfate Oxidation – Potentiometric Titration with $Fe(NH_4)_2(SO_4)_2$; Diphenylcarbazide Photometric Method; Atomic Absorption Spectrophotometry (AAS); Peroxydisulfate Oxidation – $Fe(NH_4)_2(SO_4)_2$ Reduction – $KMnO_4$ Titration; Peroxydisulfate Oxidation Amperometric Titration with $Fe(NH_4)_2(SO_4)_2$
Vanadium	Atomic Absorption Spectrophotometry; $NaHCO_3$ Hydrolysis – HNO_3 Oxidation – Potentiometric Titration with $Fe(NH_4)_2(SO_4)_2$; $Fe(NH_4)_2(SO_4)_2$ Reduction – $KMnO_4$ Titration; $KMnO_4$ – KNO_2 Urea – $Fe(NH_4)_2(SO_4)_2$
Molybdenum	Thiocyanate-$SnCl_2$ Spectrophotometric Method

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/physical-measurement-laboratory/special-publication-811 (accessed July 2017)

[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 http://www.nist.gov/srm/upload/SP260-136.PDF (accessed July 2017).

[3] 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 July 2017); 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 July 2017).

[4] Miller R.; *Simultaneous Statistical Inference*; Springer-Verlag, New York (1981).

Certificate Revision History: 19 July 2017 (Updated title; editorial changes); 20 April 2006 (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 http:/www.nist.gov/srm.