National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material® 57b

Silicon Metal

This Standard Reference Material (SRM) is intended primarily for use in evaluating chemical and instrumental methods of analysis. A unit of SRM 57b consists of a bottle containing approximately 40 g of fine powder.

Certified Mass Fraction Values: Certified values for elements in SRM 57b are provided 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 based on the results of analyses performed at NIST and collaborating laboratories.

Reference Mass Fraction Values: Reference values for constituents in SRM 57b are provided 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 [2] and are provided with associated uncertainties 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.

Information Mass Fraction Values: Information values for constituents of SRM 57b are provided 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 [2]. Information values cannot be used to establish metrological traceability.

Expiration of Certification: The certification of **SRM 57b** 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"). 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 material 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 the technical measurements for certification of this SRM was under the direction of J.R. Sieber of the NIST Chemical Sciences Division.

Measurements for value assignment of SRM 57b were performed by E.A. Mackey, A.F. Marlow, R.L. Paul, and J.R. Sieber of the NIST Chemical Sciences Division. Additional measurements were performed by R. Martin, Globe Metallurgical, Inc. (Beverly, Ohio); P. Galler, K. Blandhol, A. Storesund, A.S. Elkem, an M. Grønn, Technology Central Analytical Laboratory (Kristiansand, Norway); A. Raab, S. Freitag, and J. Feldmann, University of Aberdeen (Aberdeen, Scotland); and L. Szentmiklosi, T. Belgya, B. Fazekas, B. Maroti, G. Molnár, J. Östör, and Zs. Réva, Hungarian Academy of Sciences (Budapest, Hungary).

Statistical consultation for this SRM was provided by D.D. Leber and A.N. Heckert 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

Robert L. Watters, Jr., Director
Office of Reference Materials

INSTRUCTIONS FOR USE

To relate analytical determinations to the assigned values on this Certificate of Analysis, a minimum sample quantity of 250 mg is recommended. The powder does not require preparation prior to weighing. The material should be stored in its original container in a cool, dry location.

PREPARATION AND ANALYSIS[(1)]

The material for SRM 57b was obtained in the form of powder prepared using a typical industrial process. The material was blended and bottled at NIST. Homogeneity testing was performed at NIST using X-ray fluorescence spectrometry. Material heterogeneity was low and fit for the purpose of value assignment. Uncertainty due to material heterogeneity was accounted for in the experimental design for quantitative analysis and captured as a minor component of the overall uncertainty of each assigned value.

Certified Mass Fraction Values: The measurand is the mass fraction for each element in silicon metal listed in Table 1. The certified values are metrologically traceable to the SI unit of mass, expressed as milligrams per kilogram. Except boron, each assigned value is an unweighted mean of the results from analytical methods shown in Table 4. The uncertainty listed with the value is an expanded uncertainty about the mean, with coverage factor *k*, calculated by combining a between-method variance with a pooled, within-method variance following the ISO/JCGM Guide [3,4]. For boron, the value is the weighted mean of the individual sets of measurements estimated using a Gaussian random effects model [5–7] and the Mandel-Paule procedure [8,9]. The uncertainty listed with each value is an expanded uncertainty about the mean [10] calculated by combining within method variances with a between method variance consistent with the ISO/JCGM Guide and its Supplement [11–13]. The expanded uncertainty, U, is calculated as $U = ku_c$, where u_c is intended to represent, at the level of one standard deviation, the combined effects of between-laboratory, within-laboratory, and inhomogeneity components of uncertainty. For all elements, the coverage factor, *k*, corresponds to an approximately 95 % confidence level.

Table 1. Certified Mass Fraction Values for SRM 57b Silicon Metal

Elements	Mass Fraction (mg/kg)	Expanded Uncertainty (mg/kg)	Expansion Factor (*k*)
Aluminum (Al)	1690	220	2.0
Boron (B)	14.43	0.27	2.0
Phosphorus (P)	16.3	1.5	2.0
Titanium (Ti)	346	49	2.0
Manganese (Mn)	78.2	7.2	2.1
Iron (Fe)	3400	60	2.4
Nickel (Ni)	15.3	1.7	2.4
Zirconium (Zr)	17.8	0.6	2.1

Reference Mass Fraction Value: The measurand is the mass fraction for each element in silicon metal listed in Table 2 as determined by the analytical methods indicated in Table 4. The reference values are metrologically traceable to the SI unit of mass, expressed as milligrams per kilogram. For calcium, chromium, and copper, each value is an unweighted mean of the results from the analytical methods. The uncertainty listed with the value is an expanded uncertainty about the mean, with coverage factor *k*, calculated by combining a between-method variance with a pooled, within-method variance following the ISO/JCGM Guide [3,4]. For cobalt and vanadium, the value is the weighted mean of the individual sets of measurements estimated using a Gaussian random effects model [5–7] and the Mandel-Paule procedure [8,9]. The uncertainty listed with each value is an expanded uncertainty about the mean [10] calculated by combining within method variances with a between method variance consistent with the ISO/JCGM Guide and its Supplement [11–13]. The expanded uncertainty, U, is calculated as $U = ku_c$, where u_c is intended to represent, at the level of one standard deviation, the combined effects of between-laboratory, within-laboratory, and inhomogeneity components of uncertainty. For all constituents, the coverage factor, *k*, corresponds to an approximately 95 % confidence level.

[(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 2. Reference Mass Fraction Values for SRM 57b Silicon Metal

Elements	Mass Fraction (mg/kg)	Expanded Uncertainty (mg/kg)	Expansion Factor (*k*)
Calcium (Ca)	22.2	4.5	2.6
Cobalt (Co)	15	3	2.0
Chromium (Cr)	17.3	3.3	2.6
Copper (Cu)	17.2	5.8	2.3
Vanadium (V)	25	4	2.0

Information Mass Fraction Values: In Table 3, the values for the listed elements are provided as additional information on the composition of the material.

Table 3. Information Values for SRM 57b Silicon Metal

Elements	Mass Fraction (mg/kg)
Hydrogen (H)	120
Carbon (C)	200
Oxygen (O)	4000
Sulfur (S)	30

Table 4. Analytical Methods

Elements	Methods	Elements	Methods
H	PGAA	V	ICP-OES; PGAA; DCP-OES
B	PGAA; ICP-OES	Cr	XRF; ICP-OES
C	Combustion	Mn	XRF; PGAA; ICP-OES
O	Combustion	Fe	XRF; PGAA
Al	XRF; PGAA; ICP-OES	Co	ICP-OES; PGAA
P	RNAA; ICP-OES	Ni	XRF; ICP-OES
S	Combustion	Cu	XRF; ICP-OES
Ca	XRF; ICP-OES	Zr	XRF; ICP-OES
Ti	XRF; PGAA; ICP-OES		

Methods Key:

PGAA:	Prompt gamma-ray activation analysis performed at NIST for boron and one collaborating laboratory for boron, cobalt, hydrogen, and vanadium
ICP-OES [14]:	Inductively-coupled plasma optical emission spectrometry performed at two collaborating laboratories
Combustion:	Combustion with infrared detection performed at one collaborating laboratory
XRF:	X-ray fluorescence spectrometry with borate fusion preparation performed at NIST
RNAA:	Radiochemical neutron activation analysis performed at NIST
DCP-OES:	Direct current plasma optical emission spectrometry performed at one collaborating laboratory

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Certificate Revision History: 28 April 2015 (Revised values for B, Co, and V based on re-evaluation of original results combined with results from new analytical methods; change of reference value for B to a certified value; changes of information values for Co and V to reference values; addition of information value for H; editorial changes); 07 November 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: telephone (301) 975-2200; fax (301) 948-3730, email srminfo@nist.gov; or via the Internet at http://www.nist.gov/srm.