National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material® 360b

Zirconium (Sn-Fe-Cr) Alloy

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. A unit of SRM 360b consists of a bottle containing approximately 100 g of chips.

Certified Mass Fraction Values: Certified mass fraction values for SRM 360b 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 based on the results of analyses performed at NIST and collaborating laboratories.

Reference Mass Fraction Values: Reference mass fraction values are given 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 mass fraction values 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 360b** 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 certification of this SRM was performed by J.R. Sieber of the NIST Chemical Sciences Division.

Measurements for value assignment of SRM 360b were performed by R.L. Paul, L.J. Wood, and L.L. Yu of the NIST Chemical Sciences Division. Analyses for certification were also performed by S.A. Martin and T.A. Policke, Babcock and Wilcox, Lynchburg, VA; S. Kallmann and C.L. Maul, Ledoux and Company, Teaneck, NJ; A.D. Fryer, Oregon Metallurgical Corporation, Albany, OR; J.H Schlewitz, Teledyne Wah Chang, Albany, OR; R. Orlowski, UNC Resources, Inc., Uncasville, CT; G. Beck, D. Dorn, C. Hanson, and C. Heinke, ATI Wah Chang, Albany, OR; L. Trecani, CEZUS, Ugine, France; A. Walczewski, J. Gast, and G. Kralik, LECO Corp., St. Joseph, MI; D. Tullis and M. Smith, Westinghouse Electric Co., Ogden, UT; L. Dilks, Laboratory Testing Inc., Hatfield, PA; C. Vancura, White Horse Technical Services, Temple City, CA; K. VonScio, Perryman Co., Houston, PA; and M. Smith, ATI Metals, Natrona Heights, PA.

Carlos A. Gonzalez, Chief
Chemical Sciences Division

Gaithersburg, MD 20899
Certificate Issue Date: 20 October 2014
Certificate Revision History on Last Page

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

Statistical consultation for this SRM was provided by W.F. Guthrie 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.

INSTRUCTIONS FOR USE

To relate analytical determinations to the values on this Certificate of Analysis, a minimum sample quantity of 200 mg is recommended. Store the material in its original container in a cool, dry location. This material is not intended for frequent use as a routine quality assurance material.

PREPARATION AND ANALYSIS[(1)]

The material for this SRM was provided by Teledyne Wah Chang, Albany, OR. The material was chipped and blended at the NIST facilities in Gaithersburg, MD. Analyses for value assignments of this SRM were performed using the methods listed in Table 4.

Certified Mass Fraction Values: The measurands are the mass fractions of the elements listed in Table 1. The certified values are metrologically traceable to the SI unit of mass. The certified value for each analyte was calculated as the mean of the means from the individual methods/laboratories. 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 [3–5]. The combined uncertainty includes contributions from inhomogeneity of the element within the zirconium alloy and variability among sets of results from the test methods used by the laboratories.

Table 1. Certified Mass Fraction Values for SRM 360b Zirconium (Sn-Fe-Cr) Alloy

Elements	Mass Fraction (mg/kg)	Expanded Uncertainty (mg/kg)	Coverage Factor *k*
Boron (B)	0.191	0.033	2.0
Chromium (Cr)	1043	18	2.4
Iron (Fe)	2138	42	2.0
Nickel (Ni)	22.5	4.4	2.1
Copper (Cu)	12.5	1.7	3.2
Hafnium (Hf)	78.5	2.3	2.6
Elements	**Mass Fraction (%)**	**Expanded Uncertainty (%)**	**Coverage Factor *k***
Tin (Sn)	1.555	0.057	2.0

(1) Certain commercial equipment, instruments, or materials are identified in this document to adequately specify the technical procedures. 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.

Reference Mass Fraction Values: The measurands are the mass fractions of elements listed in Table 2, as determined by the methods indicated in Table 4. The reference values are metrologically traceable to the SI unit of mass. The reference value for each analyte was calculated as the mean of the means from the individual methods/laboratories. For all elements, except H and Co, 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 [3–5]. For hydrogen, the reference value is a weighted mean of the results from six laboratories [6]. The uncertainty is an expanded uncertainty about the mean [7] with coverage factor, $k = 2$, (approximately 95 % confidence) calculated by combining a between method variance incorporating inter-method bias with a pooled, within-method variance. For cobalt, the assigned value is based on the results from a single method performed at NIST. The uncertainty listed with the value is an expanded uncertainty about the mean, with coverage factor *k*, calculated following the ISO/JCGM Guide [3–5].

Table 2. Reference Mass Fraction Values for SRM 360b Zirconium (Sn-Fe-Cr) Alloy

Elements	Mass Fraction (mg/kg)	Expanded Uncertainty (mg/kg)	Coverage Factor *k*
Hydrogen (H)	16.01	0.70	2.0
Carbon (C)	109	13	2.4
Nitrogen (N)	45	11	3.2
Aluminum (Al)	57	11	2.0
Silicon (Si)	80	17	2.0
Phosphorus (P)	8.7	3.4	2.0
Titanium (Ti)	15.5	3.3	2.8
Manganese (Mn)	9.2	1.6	2.8
Cobalt (Co)	0.97	0.11	2.8

Information Mass Fraction Values: The information value for each constituent 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 3. Information Mass Fraction Values for SRM 360b Zirconium (Sn-Fe-Cr) Alloy

Elements	Mass Fraction (mg/kg)	Elements	Mass Fraction (mg/kg)
Oxygen (O)	1430	Niobium (Nb)	<50
Fluorine (F)	<10	Molybdenum (Mo)	<25
Magnesium (Mg)	<1	Cadmiun (Cd)	<1
Sulfur (S)	30	Antimony (Sb)	1
Chlorine (Cl)	<1	Tantalum (Ta)	<100
Vanadium (V)	<30	Tungsten (W)	<50
Zinc (Zn)	<50	Lean (Pb)	<5
Gallium (Ga)	<1	Uranium (U)	<2
Arsenic (As)	7		

Table 4. Analytical Methods Used for SRM 360b

Elements	Methods[a]	Elements	Methods[a]
H	5	Co	2
B	1, 2	Ni	1, 3
C	5	Cu	3, 4, 8, 11
N	6, 7	Zn	8
O	5	Ga	8
F	8	As	8
Mg	8	Nb	4, 8, 13
Al	4	Mo	4, 8
Si	4, 9, 12	Cd	4, 8
P	15, 16	Sn	1, 3
S	5, 8	Sb	8
Cl	8	Hf	2, 4, 8, 12
Ti	4, 8, 9	Ta	4, 8, 13
V	8	W	4, 8
Cr	1, 3	Pb	8
Mn	2, 4, 8	U	8
Fe	1, 3		

[a] Key to Methods in Table 4:

1. Prompt gamma-ray activation analysis (PGAA) at NIST
2. Inductively coupled plasma mass spectrometry (ICP-MS) at NIST
3. Inductively coupled plasma optical emission spectrometry (ICP-OES) at NIST
4. Arc spark optical emission spectrometry (AS-OES)
5. Inert gas fusion with infrared and thermal conductivity detection
6. Kjeldahl method
7. Distillation and reaction with potassium tetraiodomercurate (II)
8. Spark source mass spectrometry (SS-MS)
9. Colorimetric – ammonium molybdate
10. Colorimetric – 5-sulfosalicylic acid
11. Atomic absorption spectrophotometry (AAS)
12. Direct current plasma optical emission spectrometry (DC-OES)
13. X-ray fluorescence spectrometry (XRF)
14. Titration
15. Flame emission spectrophotometry (FES)
16. Phosphine flame ionization detection

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: http://www.nist.gov/pml/pubs/sp811/index.cfm (accessed Oct 2014).

[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, Gaithersburg, MD (2000); available at http://www.nist.gov/srm/publications.cfm (accessed Oct 2014).

[3] Hahn, G.J.; Meeker, W.Q.; *Statistical Intervals: A Guide for Practitioners*; John Wiley & Sons, Inc., New York (1991).

[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 http://www.bipm.org/utils/common/documents/jcgm/JCGM_100_2008_E.pdf (accessed Oct 2014); 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/physlab/pubs/index.cfm (accessed Oct 2014).

[5] Levenson, M.S.; Banks, D.L.; Eberhardt, K.R.; Gill, L.M.; Guthrie, W.F.; Liu, H.-k.; Vangel, M.G.; Yen, J.H.; Zang, N.F.; *An Approach to Combining Results from Multiple Methods Motivated by the ISO GUM*; J. Res. Natl. Inst. Stand. Technol., Vol. 105, pp. 571–579 (2000).

[6] DerSimonian, R.; Laird, N.; *Meta-analysis in Clinical Trials*; Control Clin. Trials, Vol. 7, pp. 177–188 (1986).

[7] Horn, R.A.; Horn, S.A.; Duncan, D.B.; *Estimating Heteroscedastic Variance in Linear Models*; J. Am. Stat. Assoc., Vol. 70, pp. 380–385 (1975).

Certificate Revision History: 20 October 2014 (Change of hydrogen information mass fraction value to reference value; editorial changes); **18 January 2013** (Revision of material and preparation descriptions due to changes in the material bottling procedure; editorial changes); **02 October 2012** (Change of hydrogen reference mass fraction value to information value; addition of oxygen information mass fraction value; editorial changes); **10 December 2009** (This revision updates the certificate to current NIST standards and reports revised assignments and values for all constituents based on re-evaluation of the original analytical results in combination with new determinations); **21 April 1986** (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.