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

Certified Mass Fraction Values: Certified mass fraction [1] values for SRM 360b are listed in Table 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 listed in Appendix A. The uncertainty listed with the value is an expanded uncertainty, $U = ku_c$, based on a 95 % confidence level [3] and is calculated according to the method in the ISO Guide [3-5].

Reference Mass Fraction Values: Reference mass fraction [1] 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 and are provided with associated uncertainties that may not include all components of uncertainty [2]. The uncertainty listed with the value is an expanded uncertainty based on a 95 % confidence level [3] and is calculated according to the method in the ISO Guide [3-5].

Information Mass Fraction Values: Information mass fraction [1] values are given in Table 3. Information values are not certified and insufficient information is available to assess adequately their uncertainties [2]. They are given to provide additional information on the matrix.

Expiration of Certification: The certification of this SRM 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"). Periodic recalibration or recertification of this SRM is not required. This 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) will facilitate notification.

Coordination of technical measurements for certification of SRM 360b was under the direction of J.R. Sieber of the NIST Chemical Sciences Division.

The technical and support aspects involved in the original preparation, certification, and issuance of SRM 360b were coordinated through the NIST Standard Reference Materials Program by R.E. Michaelis, R. Alvarez, and W.P. Reed. The overall coordination of the technical measurements leading to certification of SRM 360b was performed under the direction of J.I. Shultz, Research Associate, ASTM/NIST Research Associate Program.

Analytical measurements at NIST for certification of this SRM were performed by R.L. Paul, L.J. Wood, and L.L. Yu 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
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Robert L. Watters, Jr., Director
Office of Reference Materials

Collaborating laboratories assisting with the original characterization were S.A. Martin, Babcock and Wilcox, Naval Nuclear Fuel Division, Lynchburg, VA; S. Kallmann and C.L. Maul, Ledoux and Company, Teaneck, NJ; A.D. Fryer, Oremet Titanium, Oregon Metallurgical Corporation, Albany, OR; J.H. Schlewitz, Teledyne Wah Chang, Albany, OR; and R. Orlowski, UNC Naval Products, Division of UNC Resources, Inc., Uncasville, CT.

Additional analytical determinations for re-evaluation of SRM 360b were performed by the following laboratories: G. Beck, D. Dorn, C. Hanson, and C. Heinke, ATI Wah Chang, Albany, OR; T.A. Policke, Babcock and Wilcox, Nuclear Operations Group, Lynchburg, VA; L. Trecani, CEZUS, Ugine, France; A. Walczewski and G. Kralik, LECO Corp., St. Joseph, MI; and D. Tullis and M. Smith, Westinghouse Electric Co., Western Zirconium Plant, Ogden, UT.

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

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. Store the material in its original container in a cool, dry location.

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.

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

Constituent	Mass Fraction[(a)] (mg/kg)	Expanded Uncertainty (mg/kg)	Coverage Factor *k*
B	0.191	0.033	2.0
Cr	1043	18	2.4
Fe	2138	42	2.0
Ni	22.5	4.4	2.1
Cu	12.5	1.7	3.2
Hf	78.5	2.3	2.6
	(%)	(%)	
Sn	1.555	0.057	2.0

[(a)] The assigned value is an unweighted mean of the results from two or more 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 Guide [3,5].

[(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.

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

Constituent	Mass Fraction[(a)] (mg/kg)	Expanded Uncertainty (mg/kg)	Coverage Factor *k*
C	109	13	2.4
N	45	11	3.2
Al	57	11	2.0
Si	80	17	2.0
P	8.7	3.4	2.0
Ti	15.5	3.3	2.8
Mn	9.2	1.6	2.8
Co[(b)]	0.97	0.11	2.8

[(a)] The assigned value is an unweighted mean of the results from two or more 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 Guide [3,5].

[(b)] The assigned value for Co 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 Guide [3].

Table 3. Information Mass Fraction Values for SRM 360b Zirconium (Sn-Fe-Cr) Alloy

Constituent	Mass Fraction (mg/kg)	Constituent	Mass Fraction (mg/kg)
H	11	As	7
O	1430	Nb	<50
F	<10	Mo	<25
Mg	<1	Cd	<1
S	30	Sb	1
Cl	<1	Ta	<100
V	<30	W	<50
Zn	<50	Pb	<5
Ga	<1	U	<2

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 Jan 2013).

[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 Jan 2013).

[3] Hahn, G.J., and 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* (ISO 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 Jan 2013); 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 Jan 2013)

[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).

Certificate Revision History: **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.

APPENDIX A

Analytical Methods[a]

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

[a] Key to Methods in Table Above

1. Prompt gamma-ray activation analysis at NIST
2. Inductively coupled plasma mass spectrometry at NIST
3. Inductively coupled plasma optical emission spectrometry at NIST
4. Arc spark optical emission spectrometry
5. Combustion
6. Kjeldahl
7. Distillation and reaction with potassium tetraiodomercurate (II)
8. Spark source mass spectrometry
9. Colorimetric – ammonium molybdate
10. Colorimetric – 5-sulfosalicylic acid
11. Atomic absorption spectrophotometry
12. Direct current plasma optical emission spectrometry
13. X-ray fluorescence spectrometry
14. Titration
15. Flame emission spectrophotometry
16. Phosphine flame ionization detection