National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material® 1728

Tin Alloy (Sn-3Cu-0.5Ag)

This Standard Reference Material (SRM) is a tin solder alloy formulated from tin, copper, and silver. SRM 1728 is intended for use in the evaluation of chemical and instrumental methods of analysis and in calibration of analyses for bulk elemental composition. In addition to methods that require disk-form specimens, SRM 1728 may be used with test methods in which chips of the material are digested to prepare solutions for measurements. Each unit of SRM 1728 consists of one disk approximately 39 mm in diameter and 15 mm thick, certified to a depth of 10 mm from its original test surface.

Certified Values: Certified values for nine constituents of SRM 1728 are reported in Table 1. For all elements, values are reported as mass fractions [1]. Value assignment categories are based on the definitions of terms and modes used at NIST for 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 using instrumental and classical test methods.

Reference Values: Reference values for five 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 Values: Information values for five 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 [2].

Expiration of Certification: The certification of **SRM 1728** 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 Handling, Storage, and Use"). However, 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 technical measurements for certification of SRM 1728 was performed by J.R. Sieber of the NIST Chemical Sciences Division.

Measurements for homogeneity testing and value assignment of SRM 1728 were performed by S.E. Long, J.L. Molloy, S.A. Rabb, and J.R. Sieber of the NIST Chemical Sciences Division.

Statistical consultation for this SRM was provided by S.D. Leigh of the NIST Statistical Engineering Division.

Carlos A. Gonzalez, Chief
Chemical Sciences Division

Gaithersburg, MD 20899
Certificate Issue Date: 29 October 2013
Certificate Revision History on Last Page

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

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

INSTRUCTIONS FOR HANDLING, STORAGE, AND USE

Each disk of SRM 1728 is certified to a depth of 10 mm from the original, as-delivered, certified surface, which is the surface opposite to the side marked with the SRM number and NIST logo. The last 5 mm of the disk is not certified. NIST has created the surfaces on each disk using a machine cutting technique. If it is necessary to create a new surface finish on a disk, it is recommended to use a technique such as fly cutting or lathe cutting. Lubricants and coolants are not recommended. Abrasive grinding is not recommended because particles of abrasive may become embedded in the alloy. In addition, pressure and localized heating are likely to cause smearing of one or more constituent elements, thus altering the composition of the surface.

Store the SRM in a cool, dry location, preferably in its original container. The material is subject to superficial corrosion, and there is some possibility of microstructural changes due to recrystallization. However, it will otherwise remain stable provided adequate precautions are taken to protect it from contamination, extremes of temperature, and moisture.

To relate the results of analysis to the assigned values and associated uncertainty estimates for SRM 1728, it is recommended to use a minimum of 0.25 g of chipped material for destructive test methods. Take chips from the disk as needed; do not chip a large quantity for later use. For X-ray fluorescence spectrometry, common spectrometer designs incorporate measurement areas ≥ 2 cm^2, which is expected to be adequate for this material. For optical emission spectrometers, such as glow discharge and arc-spark designs, the use of the average of multiple measurements is recommended for a single determination. User experience indicates that four or more "burns," each ≥ 3 mm diameter, are necessary.

PREPARATION AND ANALYSIS[(1)]

The material for SRM 1728 was prepared using a chill casting procedure at Universal Scientific Laboratory Pty. Ltd., Milperra, Australia under the direction of W. Ting. Design consultation and manufacturing quality assurance were provided by C. Eveleigh of MBH Analytical, Barnet, UK. Preparation of the disks to their final size was accomplished at NIST under the direction of M. Cronise of the NIST Office of Reference Materials. The unidirectional solidification effects associated with semi-chill casting have led to segregation and heterogeneity in the portion of the disk beyond the first 10 mm.

Measurements for homogeneity testing of SRM 1728 were performed at NIST by X-ray fluorescence spectrometry and inductively coupled plasma optical emission spectrometry. The material has not been evaluated for use with techniques for microanalysis. However, preliminary measurements using an X-ray fluorescence spectrometer having a small X-ray beam (nominally 50 µm diameter) revealed the existence of small volumes within the alloy (nominally 300 µm diameter) that are enriched in either Fe or Pb.

Quantitative determinations were performed using both the solid disks and chips prepared from the certified portions of the disks. The test methods employed by NIST were the following:

Inductively coupled plasma optical emission spectrometry: Ag, As, Bi, Cd, Co, Cr, Cu, Fe, Hg, In, Ni, Pb, S, Sb, Zn

Cold vapor isotope dilution inductively coupled plasma mass spectrometry: Hg

X-ray fluorescence spectrometry: Ag, Al, As, Bi, Cd, Cr, Cu, Fe, Ni, P, Pb, S, Si, Sn, Zn

The value assignments for SRM 1728 are traceable through calibrations of the measurement processes to composition values of primary reference materials maintained by NIST and to NIST SRMs for tin and solder alloys.

[(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 1. Certified Mass Fraction Values for SRM 1728 Tin Alloy (Sn-3Cu-0.5Ag)

Constituent	Mass Fraction[a] (%)		
Ag	0.4591	±	0.0031
Cu	3.06	±	0.12

Constituent	Mass Fraction[a] (mg/kg)		
As	96	±	20
Cd	58.2	±	7.2
Fe	111	±	17
Hg	111.98	±	0.61
Ni	81.7	±	3.6
Pb	544	±	50
S	34.9	±	0.5

[a] Each certified value is a weighted mean of the results from four to five analytical methods [3,4]. The uncertainty of a certified value is an expanded uncertainty about the mean, *U*, with coverage factor 2 (approximately 95 % confidence) calculated by combining a between-method variance incorporating inter-method bias with a pooled, within-method variance following the ISO/JCGM Guide [5,6].

Table 2. Reference Mass Fraction Values for SRM 1728 Tin Alloy (Sn-3Cu-0.5Ag)

Constituent	Mass Fraction (mg/kg)		
Bi[a]	128	±	53
Co[b]	57	±	2
Cr[a]	1.2	±	0.2
In[b]	31	±	2
Sb[b]	87	±	3

[a] Each reference value is a weighted mean of the results from four to five analytical methods [3,4]. The uncertainty of a certified value is an expanded uncertainty about the mean, *U*, with coverage factor 2 (approximately 95 % confidence) calculated by combining a between-method variance incorporating inter-method bias with a pooled, within-method variance following the ISO/JCGM Guide [5,6].

[b] Each reference value is the mean of individual results from a single NIST method. The uncertainty is expressed as an expanded uncertainty, *U*, and is calculated according to the method described in the ISO/JCGM Guide [6] as $U = ku_c$, where u_c is a combined uncertainty calculated at the level of one standard deviation. The coverage factor, $k = 2$, was chosen to approximate 95 % confidence [7].

Table 3. Information Mass Fraction Values for SRM 1728 Tin Alloy (Sn-3Cu-0.5Ag)

Constituent	Mass Fraction (%)
Sn	96.3

Constituent	Mass Fraction (mg/kg)
Al	12
P	10
Si	45
Zn	156

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://physics.nist.gov/cuu/pdf/sp811.pdf (accessed Oct 2013).

[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 (2000); available at http://www.nist.gov/srm/upload/SP260-136.PDF (accessed Oct 2013).

[3] Rukhin, A.L., *Weighted means statistics in interlaboratory studies*, Metrologia, Vol. 46, pp. 323–331 (2009).

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

[5] 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).

[6] 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 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/pml/pubs/tn1297/index.cfm (accessed Oct 2013).

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

Certificate Revision History: 28 October 2013 (Instructions for use updated; editorial changes); 29 November 2010 (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; e-mail srminfo@nist.gov; or via the Internet http://www.nist.gov/srm.