National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material® 1729

Tin Alloy (97 Sn – 3 Pb)

This Standard Reference Material (SRM) is intended for use in the evaluation of chemical and instrumental methods of analysis or in calibration of analyses for bulk elemental composition. SRM 1729 is a tin solder alloy formulated from tin and lead. In addition to methods that require disk-form specimens, SRM 1729 may be used with test methods in which chips of the material are digested to prepare solutions for measurements. A unit of SRM 1729 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 Mass Fraction Values: Certified mass fraction values for three constituents of SRM 1729 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 Mass Fraction Values: Reference mass fraction values for six 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 Mass Fraction Values: Information mass fraction values for two 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 1729** 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 or register online) will facilitate notification.

Coordination of the technical measurements for certification of SRM 1729 was performed by J.R. Sieber of the NIST Chemical Sciences Division and D. Peters of The Aerospace Corporation (El Segundo, CA).

Measurements for homogeneity testing and value assignment of SRM 1729 were performed by J.L. Molloy, S.A. Rabb, and J.R. Sieber of the NIST Chemical Sciences Division. The following collaborating laboratories provided analytical results and consultation. Each laboratory provided data from SEM-EDS or microbeam X-ray fluorescence spectrometry, or both, for characterization of material homogeneity and fitness for purpose: T. Devaney, Hi-Rel Laboratories, Spokane, Washington; T. Hester, Raytheon Corporation, El Segundo, California; E. Frasco, The Aerospace Corporation, El Segundo, California.

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Statistical consultation for this SRM was provided by S.D. Leigh 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 HANDLING, STORAGE, AND USE

Store the SRM in a cool, dry location, preferably in its original container. SRM 1729 is expected to remain stable provided adequate precautions are taken to protect it from contamination, extremes of temperature, and moisture. The material is subject to superficial corrosion, and there is the possibility of microstructural changes due to recrystallization. Exposure to elevated temperatures may cause increased diffusion of the elements Pb and Zn from the bulk to the surface.

Each disk of SRM 1729 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 because 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. With the exception of the top few micrometers of the surface, the material is sufficiently homogeneous for the intended uses to a depth of 10 mm. When the user applies a new surface to a disk, that surface will change through known physical processes and the top few micrometers will become different from the bulk.

NIST has created the surface on each disk using a machine cutting technique. If it is necessary to create a new surface finish on a disk, a technique such as lathe cutting or fly cutting is recommended. Do not use lubricants and coolants. Abrasive grinding is not recommended because particles of abrasive may become embedded in the alloy. In addition, pressure and localized heating cause smearing of one or more constituent elements, thus altering the composition of the surface. Inappropriate surface preparation may be characterized by one or more changes observable using X-ray fluorescence spectrometry (XRF), including elevated amounts of Zn on the surface as a result of elevated temperature or diminished amounts of all elements except Sn on the surface as a result of smearing of Sn.

To relate the results of analysis to the assigned values and associated uncertainty estimates for SRM 1729, it is necessary to consider the test method to be used. For X-ray fluorescence spectrometry, it is recommended to measure an area ≥ 0.5 mm diameter. For microbeam XRF spectrometer designs having smaller spot sizes, it is recommended to measure multiple locations and to combine the data to simulate a larger area. 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. "Pre-burn" conditions should be employed to provide reasonable assurance that the measurements are representative of the bulk composition.

To use SRM 1729 with test methods based on the dissolution of chips of the alloy, it is recommended to use a machine tool to remove chips of the SRM to a depth of at least 2 mm from the original surface or from the current surface provided the total depth does not exceed 10 mm from the original, as-received surface of the disk. Take chips from the disk as needed; do not chip a large quantity for later use. User experience has shown that a sample mass of 0.25 g is sufficient for a single test specimen.

SRM 1729 is not certified for use with measurement techniques that measure just the outermost 10 μm or so of the surface. This statement refers to whatever is the current surface of a disk as opposed to the original surface at time of purchase. In particular, it is known that results obtained by measurements using scanning electron microscopy with energy dispersive X-ray spectrometry (SEM-EDS) or using X-ray fluorescence spectrometry and measuring the Pb M-series emission lines will not be representative of the bulk composition of SRM 1729. Measurements performed at NIST and collaborating laboratories using SEM-EDS or XRF with variable X-ray take-off angle have shown that SEM-EDS results reflect the enrichment of Pb and Zn at the surface of the alloy. It is known that Pb and Zn diffuse toward the surfaces of Sn alloys until an equilibrium is reached in which the Pb and Zn are enriched with respect to the bulk mass fractions [3,4].

PREPARATION AND ANALYSIS[1]

The material for SRM 1729 was prepared using a semi-chill casting procedure. 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 MBH Analytical with preparation of the final certified surface done at NIST under the direction of M. Cronise of the NIST Office of Reference Materials.

Homogeneity testing was performed at NIST using wavelength-dispersive X-ray fluorescence spectrometry and microbeam X-ray fluorescence spectrometry. The material has been shown to have concentration gradients near the surface. Measurement techniques that provided information from depths of 10 μm or less indicated mass fractions significantly different from the bulk composition. 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 100 μm diameter) that are enriched in either Fe, Zn, Pb, or Bi.

For quantitative analyses, the material was tested using both the solid disks and chips prepared from the certified portions of the disks. The test methods employed by NIST were X-ray fluorescence spectrometry for the elements Ag, Al, Bi, Cu, Fe, Ni, Pb, Si, Sn, and Zn and inductively coupled plasma optical emission spectrometry (ICP-OES) for the elements Bi, Cu, Fe, Ni, Pb, Sb, and Zn. The value assignments for SRM 1729 are traceable through calibrations of the measurement processes to composition values of primary reference materials maintained by NIST and to NIST reference materials for tin and solder alloys.

Table 1. Certified Mass Fraction Values for SRM 1729 Tin Alloy (97Sn-3Pb)

Constituent	Mass Fraction[a] (%)
Pb	3.11 ± 0.16
Constituent	**Mass Fraction[a] (mg/kg)**
Bi	114.7 ± 4.5
Zn	518 ± 31

[a] The certified value is a weighted mean of a set of results obtained using the two test methods listed in the certificate [5,6]. The uncertainty listed with each value is an expanded uncertainty about the mean, 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 [7,8]. The measurands are the mass fractions of the elements listed in Table 1. Metrological traceability is to the SI derived units of mass fraction (expressed as percent for major constituents and mg/kg for minor constituents).

[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 2. Reference Mass Fraction Values for SRM 1729 Tin Alloy (97Sn-3Pb)

Constituent	Mass Fraction (%)		
Sn[a]	96.9	±	0.2

Constituent	Mass Fraction (mg/kg)		
Al[a]	460	±	110
Cu[b]	24	±	14
Fe[b]	14.1	±	9.7
Ni[a]	2.2	±	0.1
Sb[a]	96.4	±	1.8

[a] The 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 [8] 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.

[b] The reference value is a weighted mean of a set of results obtained using the two test methods listed in the certificate [5,6]. The uncertainty listed with each value is an expanded uncertainty about the mean, 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 [7,8]. The measurands are the mass fractions of the elements listed in Table 2 as determined by the methods indicated in the Preparation and Analysis section of the certificate of analysis. Metrological traceability is to the SI derived units of mass fraction (expressed as percent for major constituents and mg/kg for minor constituents).

Table 3. Information Mass Fraction Values for SRM 1729 Tin Alloy (97Sn-3Pb)[a]

Constituent	Mass Fraction (mg/kg)
Ag[b]	< 75
Si	20

[a] Information values cannot be used to establish metrological traceability.

[b] This information value is an estimate of the limit of detection, L_D, of the test method employed at NIST, calculated as $L_D = 4s$ where s is an estimate of the repeatability standard deviation of measurement of a low mass fraction specimen.

REFERENCES

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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.