



National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material® C2415a

Battery Lead (UNS 52770)

This Standard Reference Material (SRM) is intended primarily for the evaluation of methods for analysis of constituent elements in lead alloys. SRM C2415a is a Pb-Sb-Sn alloy prepared by semi-chill casting. A unit of SRM C2415a consists of one disk of lead alloy approximately 40 mm diameter and at least 18 mm thick. The disk is certified to a depth of 10 mm from the as-received surface of the side opposite the label.

Certified Mass Fraction Values: Certified values for constituents of SRM C2415a are reported in Table 1 as mass fractions on an as-received basis [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.

Reference Mass Fraction Values: One reference mass fraction value is 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, not include all sources of uncertainty, or may reflect a lack of sufficient agreement among multiple analytical methods.

Information Mass Fraction Value: One information value is reported in Table 3 as a mass fraction on an as-received basis. An information value is considered to be a value that may 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 C2415a** is valid indefinitely, within the measurement uncertainty specified, provided the SRM is handled and stored in accordance with instructions given in this certificate (see "Instructions for Handling, Storage, and 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 before expiration of this certificate, NIST will notify the purchaser. Registration (see attached sheet or register online) will facilitate notification.

Coordination of material development and technical measurements for certification were performed by J.R. Sieber of the NIST Chemical Sciences Division.

Analyses leading to the certification of this SRM were performed at NIST by S.A. Rabb, and J.R. Sieber of the NIST Chemical Sciences Division and at collaborating laboratories by P. Lawson, N. Qin, and W. Ting of Universal Scientific Laboratory (Revesby, Australia), and L. Dilks and D. McCloud of Laboratory Testing, Inc. (Hatfield, Pennsylvania).

Statistical consultation for this SRM was provided by A.L. Pintar of the NIST Statistical Engineering Division.

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

Carlos A. Gonzalez, Chief
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Robert L Watters, Jr., Director
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Gaithersburg, MD 20899
Certificate Issue Date: 07 August 2014

INSTRUCTIONS FOR HANDLING, STORAGE, AND USE

It is recommended to wear gloves when handling the disk to avoid exposure to lead metal. Store the SRM in a cool, dry location, preferably in its original container. SRM C2415a 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 elements from the bulk to the surface.

The as-received disk of SRM C2415a 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 portion of the disk beyond the first 10 mm 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 certified 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.

The certified surface of an as-received disk of SRM C2415a is not ready for immediate analyses. It is necessary to create a new surface finish on a disk. A technique such as fly cutting or lathe 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 may 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 minor and trace constituents on the surface as a result of elevated temperature or contamination, and diminished amounts of all elements except Pb on the surface as a result of smearing of Pb.

To relate the results of analysis to the assigned values and associated uncertainty estimates for SRM C2415a, it is necessary to consider the test method to be used. 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. For XRF, it is recommended to measure an area ≥ 3 mm diameter. Measurements of higher energy X-rays are expected to exhibit lower variance due to heterogeneity because higher energy X-rays can be measured from greater depths in the material. Conversely, the use of particle beam excitation of X-ray emission is not appropriate with this material because the impinging particles cannot penetrate beyond the diffusion-altered surface region.

SRM C2415a 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. It is known that certain elements diffuse toward the surfaces of Pb alloys until an equilibrium is reached in which the diffusing elements are enriched with respect to the bulk mass fractions [3].

To use SRM C2415a 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 0.12 g is sufficient for a single test specimen.

PREPARATION AND ANALYSIS⁽¹⁾

The material for SRM C2415a was prepared using a semi-chill casting procedure. Design consultation, manufacturing and manufacturing quality assurance were provided by W. Ting of Universal Scientific Laboratory, Revesby, Australia.

Homogeneity assessment used all measurements by all test methods at all laboratories to evaluate effects within and among disks across the entire casting batch. Homogeneity as a function of depth with disk was included in the assessment by taking samples from a series of depths within selected disks.

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 and the elements determined using each method are listed in Table 4.

⁽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.

Certified Mass Fraction Values: The measurand is the total mass fraction for each constituent listed in Table 1. The certified values are metrologically traceable to the SI unit of mass, expressed as percent or milligrams per kilogram as indicated in the table. Each certified value is the estimate of the mean of a random effects model fitted to the data from at least three methods. The estimates and associated uncertainties are calculated using the Bayesian inference paradigm [4]. The uncertainty listed with each certified value is an interval calculated in a consistent manner with the ISO/JCGM Guide [5,6], and it expresses contributions from all recognized sources of uncertainty, including differences among analytical methods, differences among disks, dispersion of values resulting from sample preparation and replicated measurement, preparation and measurement of calibrants, analytical calibration functions, assay of primary materials, and balance calibration. The nominal coverage for each interval is 95 %.

Table 1. Certified Mass Fraction Values for SRM C2415a

| Constituent | Mass Fraction (%) | Standard Uncertainty (%) | Interval (%) |
|----------------|----------------------|-----------------------------|------------------|
| Antimony (Sb) | 2.981 | 0.072 | 2.844 to 3.125 |
| Arsenic (As) | 0.1865 | 0.0071 | 0.1729 to 0.2011 |
| Copper (Cu) | 0.1022 | 0.0022 | 0.0983 to 0.1070 |
| Tin (Sn) | 0.3058 | 0.0048 | 0.2965 to 0.3148 |
| | (mg/kg) | (mg/kg) | (mg/kg) |
| Bismuth (Bi) | 507 | 20 | 469 to 548 |
| Cadmium (Cd) | 49.7 | 5.0 | 40.9 to 60.4 |
| Nickel (Ni) | 43.6 | 4.5 | 36.1 to 52.6 |
| Selenium (Se) | 100.5 | 4.1 | 93.2 to 109.1 |
| Silver (Ag) | 76.2 | 6.1 | 65.4 to 88.8 |
| Tellurium (Te) | 103.4 | 6.5 | 90.8 to 116.2 |

Reference Mass Fraction Value: The measurand is the mass fraction value of sulfur, based on the analytical methods indicated in Table 4. The reference value is metrologically traceable to the SI unit of mass, expressed as milligrams per kilogram. The reference value is the estimate of the mean of a random effects model fitted to the data from three methods. The estimate and associated uncertainty are calculated using the Bayesian inference paradigm. The uncertainty listed with the reference value is an interval calculated in a consistent manner with the ISO/JCGM Guide [5,6], and it expresses contributions from all recognized sources of uncertainty, including differences among analytical methods, differences among disks, dispersion of values resulting from sample preparation and replicated measurement, preparation and measurement of calibrants, analytical calibration function, assay of primary materials, and balance calibration. The nominal coverage for the interval is 95 %.

Table 2. Reference Mass Fraction Value for SRM C2415a

| Constituent | Mass Fraction (mg/kg) | Standard Uncertainty (mg/kg) | Interval (mg/kg) |
|-------------|--------------------------|---------------------------------|---------------------|
| Sulfur (S) | 61 | 12 | 42 to 85 |

Information Mass Fraction Value: In Table 3, the information value reported for lead is the mean value of the results from one method performed at NIST and the difference between 100 % and the sum of the certified values in Table 1 and the reference value in Table 2.

Table 3. Information Value for SRM C2415a

| Constituent | Mass Fraction (%) |
|-------------|----------------------|
| Lead (Pb) | 96 |

Table 4. Analytical Methods Performed at NIST and Collaborating Laboratories

| Method | Constituents |
|---|---|
| Inductively coupled plasma optical emission spectrometry (ICP-OES) at NIST | Ag, As, Bi, Cd, Cu, Ni, Sb, Se, Sn, Te |
| ICP-OES at one collaborating laboratory | Ag, As, Sb, Sn |
| Inductively coupled plasma mass spectrometry (ICP-MS) at one collaborating laboratory | Bi, Cd, Cu, Ni, S, Se, Te |
| Spark source OES at one collaborating laboratory | Ag, As, Bi, Cd, Cu, Ni, S, Sb, Se, Sn, Te |
| X-ray fluorescence (XRF) spectrometry at NIST | Ag, As, Bi, Cu, Ni, Pb, S, Sb, Sn, Te |

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/index.cfm/> (accessed Aug 2014).
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- [3] Frankenthal, R.P.; Siconolfi, D.J.; *The Equilibrium Surface Composition of Tin-Lead Alloys*; Surf. Sci., Vol. 119, pp. 331–348 (1982).
- [4] Gelman, A.; Carlin, J.B.; Stern, H.S.; Dunson, D.B.; Vehtari, A.; Rubin, D.B.; *Bayesian Data Analysis*; 3rd ed., CRC Press (2014).
- [5] 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 (JCGM) (2008); available at http://www.bipm.org/utis/common/documents/jcgm/JCGM_100_2008_E.pdf (accessed July 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/pml/pubs/index.cfm> (accessed Aug 2014).
- [6] JCGM 101:2008; *Evaluation of Measurement Data – Supplement 1 to the Guide to the Expression of Uncertainty in Measurement – Propagation of Distributions Using a Monte Carlo Method*; JCGM (2008); available at http://www.bipm.org/utis/common/documents/jcgm/JCGM_101_2008_E.pdf (accessed Aug 2014).

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 at <http://www.nist.gov/srm>.