

National Institute of Advanced Industrial Science and Technology

National Metrology Institute of Japan



Reference Material Certificate

NMIJ CRM 8203-a
No. +++



Lead-Free Solder Chip (Sn96.5 Ag3 Cu0.5) (High Concentration of Pb)

This certified reference material (CRM) was produced in accordance with the NMIJ's management system and in compliance with ISO GUIDE 34:2000 and ISO/IEC 17025:2005. This CRM is intended for use in controlling the precision of analyses and validating analytical methods and instruments used in the quantitative analysis of Pb, Ag, and Cu in a tin-based lead-free solder containing Ag and Cu.

Certified Values

The certified values of Pb, Ag, and Cu in this CRM are given in the table below. This CRM should be used without being dried. The uncertainty of the certified value is the half-width of the expanded uncertainty interval calculated using a coverage factor (k) of 2, which gives a level of confidence of approximately 95 %.

Element	Certified value, mass fraction	Expanded uncertainty, mass fraction
Pb	949.2 mg/kg	12.8 mg/kg
Ag	2.994 %	0.038 %
Cu	5041 mg/kg	65 mg/kg

Analysis

Each certified value of this CRM was determined as a weighted mean of three results. The following analytical methods were used:

- 1) Hydrofluoric acid - nitric acid digestion / isotope dilution - inductively coupled plasma mass spectrometry,
- 2) Hydrofluoric acid - nitric acid digestion / inductively coupled plasma mass spectrometry, and
- 3) Sulfuric acid - nitric acid digestion / inductively coupled plasma optical emission spectrometry.

Metrological Traceability

Each certified value of this CRM is determined by more than one method including the isotope dilution – mass spectrometry, which is the primary method of measurement, with the NMIJ primary standard solutions and the NIST SRM standard solution of Ag. The certified values are traceable to the International System of Units (SI). Each certified value is determined with the NMIJ primary standard solutions, the JCSS (Japan Calibration Service System) standard solutions, or the NIST SRM standard solutions.

Indicative Values

The indicative values of several kinds of trace metals in this CRM are given in the Table below. The uncertainty of the indicative value is the half-width of the expanded uncertainty interval calculated using a coverage factor (k) of 2, which gives a level of confidence of approximately 95 %.

Element	Indicative value, mass fraction (mg/kg)	Analytical method (* <i>vide infra</i>)
Sb	1.85 ± 0.12	1)
Bi	0.60 ± 0.08	2)
In	0.45 ± 0.04	2)
Al	≤ 2.3 (LOD)	3)
As	≤ 24 (LOD)	3)
Au	≤ 5.4 (LOD)	3)
Cd	≤ 0.44 (LOD)	3)
Cr	≤ 3.2 (LOD)	3)
Fe	≤ 0.32 (LOD)	3)
Hg	≤ 1.5 (LOD)	3)
Ni	≤ 0.69 (LOD)	3)
Zn	≤ 28 (LOD)	3)

LOD: limit of detection

* Analytical method

- 1) Hydrofluoric acid - nitric acid digestion followed by anion exchange / isotope dilution - inductively coupled plasma mass spectrometry,
- 2) Sulfuric acid - nitric acid digestion followed by vaporization separation of tin(IV) bromide / graphite-furnace atomic absorption spectrometry,
- 3) Hydrofluoric acid - nitric acid digestion / matrix matching - inductively coupled plasma optical emission spectrometry.

Expiration of Certification

The certification of this CRM is valid until March 31, 2020, provided that the material remains unopened and is stored in accordance with the instructions given in this certificate.

Sample Form

This CRM is in the form of small wire chips. The small wire chips of ca. 50 g in net volume are sealed in a plastic bag and kept in a plastic bottle.

Homogeneity

The homogeneity of the metal elements in the CRM was determined by analyzing 10 bottles selected in order of bottling with the stratified random sampling method. The concentration of the elements (Pb, Ag, and Cu) was determined by the hydrofluoric acid - nitric acid digestion / internal standard - inductively coupled plasma optical emission spectrometry. The homogeneity of each element has been incorporated into the uncertainty of the certified value. This CRM is homogeneous within the range of the uncertainty of the certified values.

Instructions for Storage

This CRM should be stored at a temperature between 15 °C and 35 °C in a clean place and should not be exposed to direct sunlight.

Instructions for Use

This CRM should be used without being dried or washed. The bottles should be allowed to warm to room temperature before opening. To maintain the homogeneity, sample mass of at least 0.20 g should be used for each analysis. For the purpose of air-buoyancy correction, the density of the material can be assumed to be 7.2 g/cm³.

Precautions for Handling

Refer to the safety data sheet (SDS) on this CRM before use.

Preparation

Base materials of this CRM were melted, casted in a mold, and promptly cooled to produce billets. The billets were extruded in a step-by-step way into wires with a diameter of 1 mm, and then cut into 1 mm x 1 mm chips. The chips of 50 g in net mass were packaged in a plastic bag under vacuum.

NMIJ Analysts

The technical manager and the production manager for this CRM is A. Hioki, and the analysts are N. Nonose and A. Hioki.

Information

If substantive technical changes occur that affect the certification before the expiration of this certificate, NMIJ will notify the registered customer. Customer registration on the NMIJ Website (given below) will facilitate notification. Technical reports regarding this CRM can be obtained from the contact details given below.

Reproduction of Certificate

In reproducing this certificate, it should be clearly indicated that the document is a copy.

April 1, 2015

Ryoji Chubachi
President

National Institute of Advanced Industrial Science and Technology

If you have any questions about this CRM, please contact
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Revision history

April 1, 2015: "Metrology Management Center" was renamed to "Center for Quality Management of Metrology."