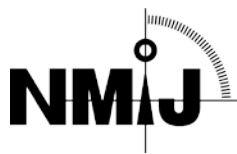


National Institute of Advanced Industrial Science and Technology

National Metrology Institute of Japan



Reference Material Certificate

NMIJ CRM 8202-a
No. +++



Lead-free Solder Chip (Sn96.5Ag3Cu0.5) (Low 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/ICE 17025:2005. It is intended for use in controlling the precision of analysis or for confirming the validity of analytical methods or instruments during the quantitative determination 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 drying. 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	197.3 mg/kg	3.3 mg/kg
Ag	3.007 %	0.036 %
Cu	5014 mg/kg	60 mg/kg

Analysis

Each certified value 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
- 3) Sulfuric acid-nitric acid digestion / inductively coupled plasma optical emission spectrometry

Metrological Traceability

Each certified value was determined by more than one method including isotope dilution / mass spectrometry as a primary method of measurement using NMIJ primary standard solutions and a NIST SRM standard solution of Ag. The certified values are traceable to the International System of Units (SI). Each information value was determined using NMIJ primary standard solutions, JCSS standard solutions, or NIST SRM standard solutions.

Indicative Values

The indicative values for several kinds of trace metals in this CRM are given in the tables 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	Information value, Mass fraction (mg/kg)	Analytical method (*shown below)
Sb	1.38 ± 0.02	1)
Bi	0.68 ± 0.16	2)
In	0.48 ± 0.05	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 methods:

- 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 homogeneities of the metal elements in the CRM were determined by analyzing 10 bottles that were selected by stratified random sampling based on the order of bottling. The concentrations of the elements (Pb, Ag, and Cu) were determined by hydrofluoric acid–nitric acid digestion / internal standard–inductively coupled plasma optical emission spectrometry. The homogeneity of each element is reflected in 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 relatively low humidity. It should be kept away from direct light.

Instructions for Use

This CRM should be used without drying or washing. The bottle should be opened after its temperature has reached room temperature. For maintaining homogeneity, the minimum recommended sample mass for analysis is 0.20 g. 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

Raw metals were melted, casted in a mold, and quickly cooled; some billets were then made. The billets were extruded stepwise into wires with a diameter of 1 mm and then cut into 1 mm chips. 50 g of chips each was packed in a plastic bag under vacuum.

NMIJ Analysts

For this CRM, the technical and production manager 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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National Metrology Institute of Japan,
Center for Quality Management of Metrology, Reference Materials Office,
1-1-1 Umezono, Tsukuba, Ibaraki 305-8563, Japan
Phone: +81-29-861-4059; Fax: +81-29-861-4009, <https://www.nmij.jp/english/service/C/>

Revision history

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