

National Institute of Advanced Industrial Science and Technology

National Metrology Institute of Japan



Reference Material Certificate

NMIJ CRM 3681-a
No. +++

Lead Isotopic Standard Solution

This certified reference material (CRM) is produced in accordance with the NMIJ's management system and is in compliance with ISO 17034 and ISO/IEC 17025. This CRM is intended as a standard for isotope ratio measurement in mass spectrometer.

Certified Values

The certified values of isotopic ratios for this CRM are given in the table below.

	Certified value, Isotopic ratio (mol/mol)	Expanded uncertainty, Isotopic ratio (mol/mol)
$^{206}\text{Pb}/^{204}\text{Pb}$	18.0900	0.0046
$^{207}\text{Pb}/^{204}\text{Pb}$	15.6278	0.0036
$^{208}\text{Pb}/^{204}\text{Pb}$	38.0626	0.0089
$^{208}\text{Pb}/^{206}\text{Pb}$	2.10406	0.00013
$^{207}\text{Pb}/^{206}\text{Pb}$	0.863888	0.000036

The isotopic abundances and molar mass calculated from the isotopic ratios are given in the tables below. The data for relative mass of lead required for the molar mass was referred from "Atomic Weights of the Elements: Review 2000" (J.R.de.Laeter et al., *Pure Appl.Chem.* **75** (2003) 683.).

	Certified value, Isotopic abundance (mol/mol)	Expanded uncertainty, Isotopic abundance (mol/mol)
^{208}Pb	0.522978	0.000016
^{207}Pb	0.214725	0.000010
^{206}Pb	0.248557	0.000009
^{204}Pb	0.013740	0.000004

	Certified value, Molar mass (g/mol)	Expanded uncertainty, Molar mass (g/mol)
Pb	207.209081	0.000028

The uncertainties of the certified values are 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 %.

Analysis

The certified values were determined by isotopic ratio measurements of $^{206}\text{Pb}/^{204}\text{Pb}$, $^{207}\text{Pb}/^{204}\text{Pb}$, $^{208}\text{Pb}/^{204}\text{Pb}$, $^{208}\text{Pb}/^{206}\text{Pb}$, $^{207}\text{Pb}/^{206}\text{Pb}$ by using multiple collector inductively coupled plasma mass spectrometer. The mass discrimination effect in the mass spectrometer was corrected by a bracketing method with a [$^{204}\text{Pb}+^{208}\text{Pb}$] mixed-enriched isotope solution, whose amount-of-substance ratio of $^{208}\text{Pb}/^{204}\text{Pb}$ was accurately determined as follows:

- (1) The ratio of total lead amount-of-substance content in a ^{204}Pb enriched isotope solution to that in a ^{206}Pb enriched isotope

one was determined by EDTA titrimetry.

- (2) The ratio obtained by (1) was corrected by metal impurities, except lead, affecting EDTA titrimetry in each enriched isotope solution determined by inductively coupled plasma sector-field mass spectrometry (ICP-SFMS).
- (3) Isotopic abundances of minor lead isotopes in each enriched isotope solution were determined by a standard addition method (ICP-SFMS) with a ^{206}Pb enriched spike solution.
- (4) A mixture of both enriched isotope solutions was made by a gravimetric preparation method, and then the amount-of-substance ratio of $^{208}\text{Pb}/^{204}\text{Pb}$ in the mixed-enriched isotope solution was corrected by the result of (3).

Metrological Traceability

The [$^{204}\text{Pb}+^{208}\text{Pb}$] mixed-enriched isotope solution used for correction of the mass discrimination effect was gravimetrically prepared from a ^{204}Pb enriched isotope solution and a ^{206}Pb enriched isotope one. The ratio of lead amount-of-substance content of both enriched isotope solutions was determined by EDTA titrimetry which is one of the primary methods of measurements. The ratio was corrected with amount of substances of metal impurities, except lead, affecting EDTA titrimetry. The metal impurities were determined by a calibration method with ICP-SFMS, where the standard solutions used for the calibrations were commercially available ones traceable to NIST SRMs. In addition, isotopic abundances of minor lead isotopes in each enriched isotope solution were determined by a standard addition method (ICP-SFMS) with a ^{206}Pb enriched spike solution, whose amount of substance of lead was determined by EDTA titrimetry with an NMIJ CRM 3608-a lead standard solution. Therefore the certified values are traceable to the International System of Units (SI).

Mutual Recognition Arrangement under Meter Convention

This certificate is consistent with the calibration and measurement capabilities (CMCs) that are included in Appendix C of the Mutual Recognition Arrangement (MRA) drawn up by the International Committee for Weights and Measures (CIPM). Under the MRA, all participating institutes recognize the validity of each other's calibration and measurement certificates for the quantities, ranges and measurement uncertainties specified in Appendix C (as for Appendix C of MRA, see <http://kcdb.bipm.org/AppendixC/default.asp>).

Indicative value

The mass fraction of lead determined by EDTA titrimetry with an NMIJ CRM 3608-a lead standard solution is given as an indicative value (unopened). Its uncertainty 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 %.

	Indicative value, Mass fraction (mg/kg)	Expanded uncertainty, Mass fraction (mg/kg)
Pb	1003.9	2.0

Expiration of Certification

This certificate is valid for three years from the date of shipment, provided that the material remains unopened and is stored in accordance with the instructions given in this certificate.

Sample Form

This CRM of ca. 100 mL in net volume is kept in a high-density polyethylene bottle with an inner lid. The bottle is sealed in an aluminum-laminated plastic bag.

Homogeneity

The homogeneity of this CRM was determined by analyzing 10 bottles selected from 235 bottles by stratified random sampling method. If the solution is mixed enough prior to use, the homogeneity within the bottle is good enough for usual purpose. The homogeneity is reflected in the uncertainties of the certified values and indicative one.

Instructions for Storage

The solution of this CRM should be kept in the high-density polyethylene bottle sealed in aluminum-laminated plastic bag.

This CRM should be stored in a clean place at a temperature of 10 °C or less without allowing it frozen.

Instructions for Use

The bottle of this CRM should be allowed to warm to room temperature before opening. Prior to use, the bottles should be shaken thoroughly but gently at room temperature.

Precautions for Handling

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

Preparation

This CRM was prepared with the same high-purity lead (mass fraction 99.999 %) as that used for an NMIJ CRM 3608-a lead standard solution. The high-purity lead was dissolved in nitric acid and diluted to 27 kg of approximately 2.5 % (mass fraction) nitric acid. The solution was divided into 100-mL bottles; each bottle contains approximately 100 mL of the solution.

Technical Information

The density of the solution used for air-buoyancy correction was 1.010 g/cm³ (25 °C).

NMIJ Analysts

The technical manager for this CRM is MIURA T., the production manager is NONOSE N., and the analysts are NONOSE N., SUZUKI T., OHATA M., ISHIZAWA Y. and HIOKI A.

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.

Note

This CRM was produced by the support of Joint Research Grant for Environmental Isotope study of Research Institute for Humanity and Nature.

April 1, 2020

ISHIMURA Kazuhiko
President

National Institute of Advanced Industrial Science and Technology

If you have any questions about this CRM, please contact:
National Institute of Advanced Industrial Science and Technology,
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://unit.aist.go.jp/nmij/english/refmate/>

Revision history

November 16, 2018: The description on "Mutual Recognition Arrangement under Meter Convention" was added.

The limit of validity of the certification was changed from "March 31, 2020" to "three years from the date of shipment"

Sample