National Research Council Canada | Conseil national de recherches Canada

Certificate of Analysis

Certified Reference Material

ELED-1

Equal atom lead isotopic standard

ELED-1 is a high purity isotopic lead Certified Reference Material (CRM). A unit of ELED-1 consists of 1 mL lead solution at 100 mg/kg in 2 % nitric acid that was prepared by mixing separated lead isotope materials to obtain approximately 1:1:1:1 ratio of lead-204, lead-206, lead-207, and lead-208. This material is intended as an isotopic standard of lead. Certified values for the isotopic composition and the atomic weight of lead have been established for this CRM. These values are listed in the Table 1. Certified values are based on measurements carried out at the National Research Council Canada (NRC). The expanded uncertainty (U_{CRM}) in the certified values is equal to ku_c where u_c is the combined standard uncertainty calculated according to the JCGM Guides [1] and protocols [2] and k is the coverage factor. A coverage factor of two (2) was applied which corresponds to approx. 95 % confidence.

Table 1: Certified quantity values and expanded uncertainties (k=2) of atomic weight, isotopic abundances, and isotope ratios of lead in ELED-1

Quantity	Value	Expanded uncertainty	Unit
Atomic weight of lead, A_r(Pb)	206.2450	0.0004	1
Isotopic abundance, $x(^{204}Pb)$	0.246 75	0.000 12	mol/mol
Isotopic abundance, $x(^{206}Pb)$	0.251 75	0.000 06	mol/mol
Isotopic abundance, $x(^{207}Pb)$	0.239 58	0.000 08	mol/mol
Isotopic abundance, $x(^{208}Pb)$	0.261 92	0.000 11	mol/mol
Isotope ratio, $n(^{204}Pb)/n(^{206}Pb)$	0.980 15	0.000 59	mol/mol
Isotope ratio, $n(^{207}Pb)/n(^{206}Pb)$	0.951 67	0.000 36	mol/mol
Isotope ratio, $n(^{208}Pb)/n(^{206}Pb)$	1.040 43	0.000 52	mol/mol
Isotope ratio, $n(^{204}Pb)/n(^{207}Pb)$	1.029 92	0.000 72	mol/mol
Isotope ratio, $n(^{206}Pb)/n(^{207}Pb)$	1.050 78	0.000 40	mol/mol
Isotope ratio, $n(^{208}Pb)/n(^{207}Pb)$	1.093 27	0.000 69	mol/mol

Isotopic composition of lead (Table 1) was determined by multi-collector inductively-coupled plasma mass spectrometry (MC-ICPMS) using two independent methods of calibration: (1) the gravimetric isotope mixture method based on the use of four high-purity isotopes of lead and (2) the regression method with NIST SRM 997 thallium isotopic standard as the calibrator. Both calibration methods provide consistent results [3].

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Atomic masses of lead isotopes are taken from 2016 Atomic Mass Evaluation [4]. The molar mass, M(Pb), and the atomic weight of lead, A_r(Pb), are related as M(Pb) = A_r(Pb)M_u, where M_u is the molar mass constant, M_u = 1 g/mol with negligible uncertainty.

Certified values

Certified values are considered to be those for which the NRC has the highest confidence in accuracy and that all known and suspected sources of bias have been taken into account and are reflected in the stated expanded uncertainties. Certified values are the best estimate of the true value and uncertainty.

Intended use

This certified reference material is intended to be used as the isotopic standard of lead with special focus on the lead isotope analysis using the double spike technique.

Storage

It is recommended that the material is stored at room temperature and the vials only opened immediately prior to use in a clean area with precautions taken against contamination.

Preparation of material

The reference material was prepared from separated lead isotopes in metallic form. The chemical purity of the lead-206 material was determined by glow discharge mass spectrometry (GDMS) at the NRC [5] whereas the purity of other isotopes was determined by high-resolution ICPMS giving the following chemical purity (mass fraction of lead): 0.999 43 kg/kg (lead-204), 0.999 95 kg/kg (lead-206), 0.999 54 kg/kg (lead-207), and 0.999 85 kg/kg (lead-208) [3]. Afterward, the lead isotopes were cleaned with 5 % HNO_3, rinsed with water, and dried in a class 10 fume hood before their quantitative dissolution in 30 % HNO_3. Appropriate aliquots of stock solutions were then weighed and mixed to obtain approximately 1:1:1:1 ratio of lead-204, lead-206, lead-207, and lead-208 in the final solution. The solutions were bottled in 4 mL acid pre-cleaned plastic bottles. Each vial contains 1 mL solution with mass fraction lead approx. at 100 mg/kg in 2 % HNO_3.

Stability

Potential instabilities due to long-term storage and transport were considered, and such effects deemed to be negligible on the isotopic composition of the material. The material is deemed stable with respect to the certified values for ten years.

Homogeneity

The material is deemed to be homogeneous with respect to the isotopic composition of lead.

Uncertainty

The overall combined uncertainty estimate includes the uncertainties in the batch characterization which include the primary standards, calibration model, and measurement repeatability.

Metrological traceability

Isotopic composition results presented in this certificate are traceable to the SI through two independent calibration methods. The gravimetric mixture method involves the purity assessment of four lead isotope materials available in metallic form and gravimetrically preparing 1:1 mixtures of them in pairs [3,5]. These isotope mixtures serve to calibrate the MC-ICPMS response. Independently, the regression method with NIST SRM 997 thallium isotopic standard [6,7] was also used to calibrate the MC-ICPMS response [8]. As such, ELED-1 serves as a suitable reference material for laboratory quality assurance programs, as outlined in ISO/IEC 17025.

Quality Management System (ISO 17034, ISO/IEC 17025)

This material was produced in compliance with the NRC Metrology Quality Management System, which conforms to the requirements of ISO 17034 and ISO/IEC 17025. The Metrology Quality Management System supporting NRC Calibration and Measurement Capabilities, as listed in the *Bureau international des poids et mesures* (BIPM) Key Comparison Database (kcdb.bipm.org/), has been reviewed and approved under the authority of the Inter-American Metrology System (SIM) and found to be in compliance with the expectations of the *Comité international des poids et mesures* (CIPM) Mutual Recognition Arrangement. The SIM approval is available upon request.

Updates

Users should ensure that the certificate they have is current. Our website at www.nrc.gc.ca/crm will contain any new information.

References

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Cited by

A list of scientific publications citing ELED-1 can be found at doi.org/10.4224/crm.2021.eled-1.

Authorship

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This Certificate is only valid if the corresponding material was obtained directly from the NRC or an Authorized Reseller.

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