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Certificate of Analysis

Certified Reference Material

HIPB-1

High Purity Lead Certified Reference Material for Lead Mass Fraction, Atomic Weight, Isotopic Composition, and Elemental Impurities

HIPB-1 is a high purity lead Certified Reference Material (CRM). A unit of HIPB-1 consists of approx. 1 g of high purity lead wire. This material is intended as a primary standard for the determination of mass fraction of lead and as an isotopic standard of lead.

Certified values for the mass fraction, atomic weight, and isotopic composition of lead, as well as mass fractions of elemental impurities have been established for HIPB-1 CRM. These values are listed in the Tables 1-3. 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 $U = ku_c$ where u_c is the combined standard uncertainty calculated according to the JCGM Guides [1] and protocols [2-3], and k is the coverage factor. A coverage factor of two (2) was applied. It is intended that U_{CRM} accounts for every aspect that reasonably contributes to the uncertainty of the measurement.

Table 1: Certified quantity values and expanded uncertainties (k=2) of mass fraction, atomic weight, and isotopic abundances of lead in HIPB-1

Quantity	Value	Expanded uncertainty
Mass fraction of lead, w(Pb)/(kg/kg)	0.999 990	0.000 002
Atomic weight of lead, A_r(Pb)	207.1791	0.0002
Isotopic abundance, $x(^{204}\mathrm{Pb})$/(mol/mol)	0.012 822	0.000 012
Isotopic abundance, $x(^{206}\mathrm{Pb})$/(mol/mol)	0.270 96	0.000 08
Isotopic abundance, $x(^{207}\mathrm{Pb})$/(mol/mol)	0.203 511	0.000 026
Isotopic abundance, $x(^{208}\mathrm{Pb})$/(mol/mol)	0.512 71	0.000 10

Table 1 shows derived quantities that characterize the properties of the lead in HIPB-1, and these quantities were calculated using the following mathematical expressions:

$$w(\mathrm{Pb}) = 1 - \sum_{E} w(\mathrm{E})$$

$$A_r(\mathrm{Pb}) = \sum_{q} A_r(^{q}\mathrm{Pb}) \cdot x(^{q}\mathrm{Pb})$$

$$x(^{A}\mathrm{Pb}) = \frac{n(^{A}\mathrm{Pb})/n(\mathrm{Pb})}{\sum_{q} n(^{q}\mathrm{Pb})/n(\mathrm{Pb})}$$

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Here E refers to the elements listed in Table 3. Other elements such as hydrogen, noble gases or any other elements not listed in Table 3 were not considered in the purity estimate. The *q* refers to all stable lead isotopes, and $A_r(^qPb)$ are the atomic weights of lead isotopes which derive from the atomic masses as listed in the 2016 Atomic Mass Evaluation [4]. The molar mass (M) and the atomic weight (A_r) of lead 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.

Tables 2-3 show the measured quantities in HIPB-1. Isotopic composition of lead (Table 2) was determined by multi-collector ICPMS using full gravimetric isotope mixture and regression methods for calibration [5,6]. Elemental impurities of lead (Table 3) were determined by GDMS using measurement models and methods with traceability to the SI through a network of CRMs [3,7]. For the purposes of obtaining the mass fraction (purity) estimate of lead, all elemental impurities below detection limit were interpreted as half the detection limit (for example '<2 µg/kg' for selenium is interpreted as 1 µg/kg). Robust estimator (median) was used to summarize the observed values from 15 units. These findings are reported in Table 3.

Table 2: Certified quantity values and expanded uncertainties (*k*=2) of lead isotope ratios in HIPB-1

Quantity	Value	Expanded uncertainty
Isotope ratio, $n(^{206}Pb)/n(^{204}Pb)$	21.133	0.014
Isotope ratio, $n(^{207}Pb)/n(^{204}Pb)$	15.873	0.014
Isotope ratio, $n(^{208}Pb)/n(^{204}Pb)$	39.99	0.04
Isotope ratio, $n(^{204}Pb)/n(^{206}Pb)$	0.047 319	0.000 030
Isotope ratio, $n(^{207}Pb)/n(^{206}Pb)$	0.751 07	0.000 22
Isotope ratio, $n(^{208}Pb)/n(^{206}Pb)$	1.8922	0.0010
Isotope ratio, $n(^{204}Pb)/n(^{207}Pb)$	0.063 00	0.000 06
Isotope ratio, $n(^{206}Pb)/n(^{207}Pb)$	1.3314	0.0004
Isotope ratio, $n(^{208}Pb)/n(^{207}Pb)$	2.5193	0.0008
Isotope ratio, $n(^{204}Pb)/n(^{208}Pb)$	0.025 010	0.000 026
Isotope ratio, $n(^{206}Pb)/n(^{208}Pb)$	0.528 49	0.000 28
Isotope ratio, $n(^{207}Pb)/n(^{208}Pb)$	0.396 94	0.000 12

Table 3: Certified quantity values and expanded uncertainties (*k*=2) of mass fractions of elemental impurities in HIPB-1 (µg/kg)

Impurity element, E	Mass fraction, *w*(E)	Expanded uncertainty	Impurity element, E	Mass fraction, *w*(E)	Expanded uncertainty
Li	<0.06	0.07	Pd	12	5
Be	<0.04	0.02	Ag	5330	1600
B	<0.1	0.2	Cd	2070	600
C	25	300	In	0.7	1.5
N	3	30	Sn	6	3
O	53	500	Sb	0.5	0.4

Impurity element, E	Mass fraction, *w*(E)	Expanded uncertainty	Impurity element, E	Mass fraction, *w*(E)	Expanded uncertainty
F	<0.6	1.5	Te	<0.3	0.2
Na	12	100	I	<0.07	0.18
Mg	<0.1	1.5	Cs	<0.09	0.13
Al	1	25	Ba	<0.1	0.1
Si	0.4	2.1	La	<0.03	0.02
P	<0.09	0.24	Ce	<0.02	0.01
S	5	5	Pr	<0.1	0.3
Cl	33	170	Nd	<0.08	0.4
K	<6	7	Sm	<0.6	1.5
Ca	<2	25	Eu	<0.3	0.8
Sc	<0.05	0.04	Gd	<0.5	1.3
Ti	<0.05	0.41	Tb	<0.1	0.3
V	<0.04	0.16	Dy	<9	23
Cr	<0.07	0.04	Ho	<0.1	0.3
Mn	<0.07	0.04	Er	<0.4	1
Fe	0.4	1	Tm	<0.1	0.3
Co	<0.05	0.03	Yb	<0.4	1
Ni	32	6	Lu	<0.1	0.3
Cu	170	90	Hf	<0.1	0.1
Zn	7	4	Ta	<240	400
Ga	<0.3	0.2	W	<0.2	0.1
Ge	<0.6	0.6	Re	<0.2	0.3
As	0.5	0.3	Os	<0.7	1
Se	<2	1	Ir	1	3
Br	<0.8	2	Pt	13	4
Rb	<0.1	0.1	Au	<32	30
Sr	<0.04	0.03	Hg	<14	11
Y	<0.04	0.06	Tl	580	120
Zr	<0.06	0.03	Pb	Matrix	Matrix
Nb	<0.03	0.02	Bi	1200	360
Mo	<0.3	0.2	Th	<0.06	4
Ru	<0.5	0.7	U	<0.06	0.09
Rh	<32	50			

Supplementary data

The accompanying datasheets (available from doi.org/10.4224/crm.2020.hipb-1) provide elemental impurity results from the analyzed CRM units, the mass and chemical amount of lead in any given unit along with the mass of individual units and their expanded uncertainties. The serial number corresponding to each unit is located on the HIPB-1 glass vial.

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 dissolved, in whole, when making a primary standard for the determination of the mass fraction of lead and as the isotopic standard of lead. If the procedure for preparing the primary standard solution involves weighing of the CRM, an appropriate cleaning should be performed to remove surface contamination.

Mass fractions of the impurities reported on this certificate are not intended for calibration purposes. This data presented simply to allow users to compute/derive purity and assess the impact of concomitant impurities when a mixed element standard solution is prepared. However, if the impurity values are used in a measurement (XRF, laser ablation etc.) appropriate care should be taken to remove any surface contamination.

Storage

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

Preparation of material

The reference material was prepared from a high-purity metallic lead wire of 2.4 mm diameter. It was cut into one-gram pieces of 22 mm length using ceramic cutter at the NRC and bottled in 4 mL glass vials filled with argon.

Stability

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

Homogeneity

The material was tested for homogeneity at the NRC by analyzing 15 CRM units. See doi.org/10.4224/crm.2020.hipb-1 for data on each of these units.

Uncertainty

The overall combined uncertainty estimate includes the uncertainties in the batch characterization and uncertainties related to possible between-unit variation (homogeneity). Sources of uncertainty considered for batch characterization include the primary standards, calibration model, and measurement repeatability.

Metrological traceability

Isotopic composition results presented in this certificate are traceable to the SI through gravimetric weighing [5] and NIST SRM 997 thallium isotopic standard [8,9]. Results of the elemental impurities and the mass fraction of lead (purity) presented in this certificate are traceable to the SI through a network of CRMs [3,7] supported by international measurement intercomparisons. As such, HIPB-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 HIPB-1 can be found at doi.org/10.4224/crm.2020.hipb-1.

Authorship

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HIPB-1

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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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