Bundesanstalt für Materialforschung und -prüfung

Certificate for Primary Reference Material

BAM-Y004

high purity lead

(BAM-A-primary-Pb-1)

Certified quantity value

When applying the prescribed sample treatment as specified below, the mass fraction *w* of lead in material BAM-Y004 is:

Element	Mass fraction *w* in kg/kg	Uncertainty in kg/kg
Lead	**0,999 92**	**± 0,000 06**

The uncertainty given is an expanded uncertainty $U=k \cdot u_c$ with $k=2$ evaluated according the GUM [1].

Taking the value for the atomic weight of lead to be (207,177 88 ± 0,000 07) as measured in the Isotopic Measurement Laboratory at BAM, the certified value corresponds to a lead amount-of-substance content of k(Pb, BAM-Y004) $=w/M=$ (4,826 37 ± 0,000 29) mol/kg (expanded uncertainty $U=k \cdot u_c$ with $k=2$) in the solid material.

The certified value has been established by subtracting the sum of the mass fractions of 91 possible impurities with other elements from the value for ideal purity of 1,000 000 kg/kg.
72 of 91 values of possible impurities with other elements are based on measurements. The sum of the mass fractions of the measured impurities is w_Σ(measured values) = (28 ± 11) mg/kg.
From theoretical considerations very reasonable estimates have been made for the upper limits of the impurities with noble gases and artificial/instable elements. The sum of the mass fractions of these 12 of 91 possible impurities is w_Σ(theoretical values) = (0,26 ± 0,25) mg/kg. The influence of this value on the certified value and its associated uncertainty is marginal.
The impurities of H, P, Si, F, Cl, Br and I (7 of 91 possible impurities) have not been measured yet. For these impurities upper limit values have been conservatively estimated. The sum of the mass fractions of the impurities estimated is w_Σ(estimated values) = (55 ± 28) mg/kg. The influence of this value on the certified value and its associated uncertainty is significant.

Note: In line with the ISO 31-0 (1992) in this document the comma (and not the dot) is used as a decimal separator.

Note, that the values for w_Σ(measured values), w_Σ(theoretical values) and w_Σ(estimated values) are not certified but only of informative nature. The uncertainties given for these three values are combined uncertainties u_c.

Safety instructions: Avoid skin contact and incorporation. Handle with caution - as any chemical substance.

Sample description: The material is provided in glass bottles containing about 0,5 g of compact material.

Recommendation for correct storing: The bottle should be kept closed at ambient conditions and in a clean and dry environment.

Expiration of certificate: This certificate expires formally ten years after seal affixation on the certificate. (The material itself is assumed to be stable > 200 years).

Prescribed sample treatment

Before use, the material has to be etched in order to achieve the defined state, to which the certified quantity value applies. Solvents of high purity (at least p.a. grade) must be used. The material must be processed (i.e. weighed) within 1 h after the final etching.

T1. The preparation has to be performed under clean air conditions and using high purity acids, high purity water and purified vessels.

T2. Sub-sample masses of $m > 0{,}2$ g and a surface to mass ratio $A/m < 14$ cm^2/g must be used.

T3. Etch individual metal pieces for 45 s in 20 mL (98 % acetic acid + 30 % H_2O_2) (3 Vol. + 1 Vol.) while moving the solution at room temperature. Use the solution only once. Rinse in 3 × 50 mL H_2O (in an ultrasonic bath).

T4. Rinse 3× in CH_3OH; expose 1 min to hair drier or 10 min in Ar-flow.

T5. The sample can then mechanically be cut using clean cutting tools. Avoid surface contamination and re-oxidation.

T6. Process material again as described in T3 and T4. The material must then have a shiny metallic surface, free of white spots.

Obey the safety instructions of the chemicals and equipment used!

Notes

N01. The certified quantity value with associated measurement uncertainty carried by BAM-Y004, is traceable to the related units of the Système International d'Unités (SI).

N02. The certified value with associated measurement uncertainty carried by BAM-Y004, is a realisation of the unit for mass fraction measurements of lead. Using the concept of molar mass and gravimetry, it is a realisation of the unit for amount-of-substance content (amount-of-substance per mass) measurements of lead.

N03. In co-operation with Physikalisch Technische Bundesanstalt (PTB), BAM-Y004 is the primary National Measurement Standard of Germany for the measurement of lead.

N04. BAM-Y004 is intended to be used to prepare gravimetrically secondary measurement standards (solid or solution).

N05. The material is intended for metrological use within National and International Metrology Institutes only.

N06. Technical details about the certification can be found in the 'Certification Report BAM-Y004', which is available from BAM on request and at www.bam.de.

N07. The certified value with associated measurement uncertainty has been established by measuring the impurities in a high purity lead material using a variety of measurement methods. The quantity values for the individual impurities are given as additional information in Table 1. These quantity values are not certified values.

N08. All measurements, which were used for the certification of BAM-Y004 have been performed by BAM laboratories.

Abbreviations

HE	Carrier gas hot extraction and combustion analysis
HR-ICP-MS	High resolution mass spectrometry using inductively coupled plasma
ICP-MS	Mass spectrometry using inductively coupled plasma
THE	Estimated value based on theoretical considerations
U	Expanded measurement uncertainty
u_c	Combined measurement uncertainty
ULV	Upper limit value based on measurements
ULV-EST	Upper limit value based on estimate; not yet measured
unc. contr.	Parameter characterising the relative influence of the uncertainty of an individual impurity mass fraction to the combined uncertainty of the lead mass fraction calculated according to: $u^2(w_i)/\sum u^2(w_j)$

Table 1: The individual values used to establish the certified value. Note, that the values given here are not certified, but only of informative nature!

	method		*w* mg/kg	*u(w)* mg/kg	unc. contr.		method		*w* mg/kg	*u(w)* mg/kg	unc. contr.		method		*w* mg/kg	*u(w)* mg/kg	unc. contr.
H	ULV-EST	<	50	25	0,69	Ge	ICP-MS	<	0,05	0,025	<< 0,01	Eu	ICP-MS	<	0,001	0,0005	<< 0,01
He	THE	<	0,001	0,0005	<< 0,01	As	ICP-MS	<	0,05	0,025	<< 0,01	Gd	ICP-MS	<	0,001	0,0005	<< 0,01
Li	ICP-MS	<	0,1	0,05	<< 0,01	Se	ICP-MS	<	1	0,5	<< 0,01	Tb	ICP-MS	<	0,001	0,0005	<< 0,01
Be	ICP-MS	<	0,1	0,05	<< 0,01	Br	ULV-EST	<	10	5	0,03	Dy	ICP-MS	<	0,001	0,0005	<< 0,01
B	ICP-MS	<	1	0,5	<< 0,01	Kr	THE	<	0,001	0,0005	<< 0,01	Ho	ICP-MS	<	0,001	0,0005	<< 0,01
C	HE	<	16	8	0,07	Rb	ICP-MS	<	0,01	0,005	<< 0,01	Er	ICP-MS	<	0,001	0,0005	<< 0,01
N	HE	<	6	3	0,01	Sr	ICP-MS	<	0,001	0,0005	<< 0,01	Tm	ICP-MS	<	0,001	0,0005	<< 0,01
O	HE	<	12	6	0,04	Y	ICP-MS	<	0,005	0,0025	<< 0,01	Yb	ICP-MS	<	0,001	0,0005	<< 0,01
F	ULV-EST	<	10	5	0,03	Zr	ICP-MS	<	0,001	0,0005	<< 0,01	Lu	ICP-MS	<	0,001	0,0005	<< 0,01
Ne	THE	<	0,001	0,0005	<< 0,01	Nb	ICP-MS	<	0,001	0,0005	<< 0,01	Hf	ICP-MS	<	0,001	0,0005	<< 0,01
Na	ICP-MS	<	0,7	0,35	<< 0,01	Mo	ICP-MS	<	0,01	0,005	<< 0,01	Ta	ICP-MS	<	0,001	0,0005	<< 0,01
Mg	ICP-MS	<	0,05	0,025	<< 0,01	Tc	THE	<	0,001	0,0005	<< 0,01	W	ICP-MS	<	0,05	0,025	<< 0,01
Al	ICP-MS	<	0,5	0,25	<< 0,01	Ru	ICP-MS	<	0,01	0,005	<< 0,01	Re	ICP-MS	<	0,001	0,0005	<< 0,01
Si	ULV-EST	<	10	5	0,03	Rh	ICP-MS	<	0,01	0,005	<< 0,01	Os	ICP-MS	<	0,001	0,0005	<< 0,01
P	ULV-EST	<	10	5	0,03	Pd	ICP-MS	<	0,01	0,005	<< 0,01	Ir	ICP-MS	<	0,005	0,0025	<< 0,01
S	HE	<	10	5	0,03	Ag	ICP-MS		0,34	0,102	<< 0,01	Pt	ICP-MS	<	0,05	0,025	<< 0,01
Cl	ULV-EST	<	10	5	0,03	Cd	ICP-MS	<	0,01	0,005	<< 0,01	Au	ICP-MS	<	0,2	0,1	<< 0,01
Ar	THE	<	0,5	0,25	<< 0,01	In	ICP-MS	<	0,005	0,0025	<< 0,01	Hg	ICP-MS	<	0,2	0,1	<< 0,01
K	ICP-MS	<	0,5	0,25	<< 0,01	Sn	ICP-MS	<	0,1	0,05	<< 0,01	Tl	ICP-MS	<	0,2	0,1	<< 0,01
Ca	ICP-MS	<	0,5	0,25	<< 0,01	Sb	ICP-MS	<	0,005	0,0025	<< 0,01	Pb			matrix		
Sc	ICP-MS	<	0,3	0,15	<< 0,01	Te	ICP-MS	<	0,02	0,01	<< 0,01	Bi	ICP-MS	<	2	1	<< 0,01
Ti	ICP-MS	<	0,05	0,025	<< 0,01	I	ULV-EST	<	10	5	0,03	Po	THE	<	0,001	0,0005	<< 0,01
V	ICP-MS	<	0,05	0,025	<< 0,01	Xe	THE	<	0,001	0,0005	<< 0,01	At	THE	<	0,001	0,0005	<< 0,01
Cr	ICP-MS	<	0,05	0,025	<< 0,01	Cs	ICP-MS	<	0,001	0,0005	<< 0,01	Rn	THE	<	0,001	0,0005	<< 0,01
Mn	ICP-MS	<	0,05	0,025	<< 0,01	Ba	ICP-MS	<	0,003	0,0015	<< 0,01	Fr	THE	<	0,001	0,0005	<< 0,01
Fe	ICP-MS	<	0,2	0,1	<< 0,01	La	ICP-MS	<	0,001	0,0005	<< 0,01	Ra	THE	<	0,001	0,0005	<< 0,01
Co	ICP-MS	<	0,02	0,01	<< 0,01	Ce	ICP-MS	<	0,001	0,0005	<< 0,01	Ac	ICP-MS	<	0,001	0,0005	<< 0,01
Ni	ICP-MS		0,74	0,222	<< 0,01	Pr	ICP-MS	<	0,001	0,0005	<< 0,01	Th	ICP-MS	<	0,001	0,0005	<< 0,01
Cu	ICP-MS		0,27	0,081	<< 0,01	Nd	ICP-MS	<	0,005	0,0025	<< 0,01	Pa	ICP-MS	<	0,001	0,0005	<< 0,01
Zn	ICP-MS	<	0,2	0,1	<< 0,01	Pm	THE	<	0,001	0,0005	<< 0,01	U	ICP-MS	<	0,001	0,0005	<< 0,01
Ga	ICP-MS	<	0,3	0,15	<< 0,01	Sm	ICP-MS	<	0,005	0,0025	<< 0,01	Value contributes >10 % to combined uncertainty					

References

1 Guide to the Expression of Uncertainty in measurement, ISO, Geneve, (1993).

BAM Berlin
Department I
Analytical Chemistry; Reference Materials

p.p.

Prof. Dr. U. Panne
(Head of Department)

BAM Berlin
Division I.1
Inorganic Chemical Analysis; Reference Materials
p.p.

Dr. N. Jakubowski
(Head of Division)

Berlin,

for further information please contact:
Federal Institute for Materials Research and Testing (BAM); Department I; D-12000 Berlin, Germany
Tel.: +49 30 8104 1116 Fax.: +49 30 8104 1117 e-mail: heinrich.kipphardt@bam.de