BAM
Bundesanstalt für Materialforschung und -prüfung

CERTIFICATE

CERTIFIED REFERENCE MATERIAL BAM-M113

Lead alloy PbCaSn

Certified Value(s)

Element	Mass fraction [1] in %	Uncertainty [2] in %
Ca	0.124	0.005
Sn	1.047	0.019
Bi	0.0194	0.0008
Al	0.0145	0.0009
	in mg/kg	**in mg/kg**
Ag	64.7	1.5
Cu	18.9	0.8
Fe [3]	1.0	0.5
Sb	5.4	1.0

1) Unweighted mean value of the means of accepted sets of data (consisting of at least 3 single results), each set being obtained by a different laboratory and/or a different method of measurement.

2) Estimated expanded uncertainty *U* with a coverage factor of $k = 2$, corresponding to a level of confidence of approx. 95 %, as defined in the Guide to the Expression of Uncertainty in Measurement, (GUM, ISO/IEC Guide 98-3:2008).

3) The mean value is estimated using the marginal likelihood for the mean (see report)

Element	Mass fraction (limits) [4] in mg/kg	Uncertainty [5] (error probability)
As	< 1	0.05
Cr	< 0.5	0.05
Mn	< 0.5	0.05
Se	< 1	0.05

4) The upper limit is estimated by calculating the 95% quantile of the marginal likelihood distribution (see report)

5) The uncertainty refers to the probability of errors or in other words the significance level. A commonly used significance level is 0.05, which means that the probability for an error (i.e., the true value is outside of the given range) is 5%,

End of Validity

This certificate is valid until there is a revocation from the producer of the material.

Material Description

The Reference Material is available in the form of discs (approx. 38 mm diameter and 38 mm height).

Recommended Use

The CRM is intended for establishing or checking the calibration of spark optical emission spectrometers for the analysis of samples of similar matrix composition. The minimum sample size for wet chemical analysis is 0.2 g.

Handling

Before use, the surface of the material must be prepared by milling or turning on a lathe. For wet chemical analysis chips must be prepared by turning or milling of the sample surface.

Transport and Storage

The material should be stored in a dry and clean environment at room temperature. Transport can be done under normal ambient conditions.

Metrological Traceability

To ensure traceability of the certified mass fractions to the SI (Système International d'Unités) calibration was performed using standard solutions prepared from pure metals or stoichiometric compounds or with traceable commercial calibration solutions.

Means of Accepted Data Sets

Certified values

Mass fraction in % | Mass fraction in mg/kg

Line No.	Ca	Sn	Bi	Al		Ag	Cu	Fe	Sb	As	Cr	Mn	Se
1	---	---	0.0173	0.0119		60.0	17.0	---	---	< 0.18	< 0.1	0.02	< 0.3
2	0.1187	---	0.0180	0.0130		62.8	17.2	0.39	---	< 0.2	< 0.1	0.02	< 0.44
3	0.1195	1.020	0.0187	0.0142		63.3	17.4	0.52	4.0	< 0.32	0.15	< 0.1	< 0.5
4	0.1202	1.021	0.0191	0.0143		64.1	17.7	0.67	4.6	< 0.5	0.22	0.1	< 0.7
5	0.1212	1.023	0.0197	0.0145		64.2	18.8	0.83	5.4	< 1	0.22	0.11	0.94
6	0.1222	1.042	0.0200	0.0146		64.4	19.2	< 1	5.5	---	< 0.5	< 0.5	< 1
7	0.1229	1.042	0.0201	0.0147		65.1	19.5	< 1	5.8	< 2	< 1	< 1	< 2
8	0.1229	1.043	0.0202	0.0148		65.4	19.5	1.43	7.4		< 1	< 1	
9	0.1253	1.045	0.0206	0.0149		66.6	19.6	1.50					
10	0.1277	1.045	0.0209	0.0150		66.8	19.9	1.63					
11	0.1277	1.073		0.0162		68.9	20.3	1.89					
12	0.1320	1.079		0.0162		---	21.2						
13	0.1322	1.087											
M	0.1243	1.047	0.0194	0.0145		64.7	18.9	0.97*	5.4	< 1**	< 0.5**	< 0.5**	< 1**
s_M	0.0046	0.024	0.0012	0.0012		2.4	1.4	0.79*	1.2				
$\bar{s}_i$	0.0025	0.019	0.0004	0.0008		1.1	0.6	0.2	0.6				

The laboratory mean values have been examined statistically to eliminate outlying values. Where a " --- " appears in the table it indicates that an outlying value has been omitted. A data set consists of at least 3 but usually 6 single values of one laboratory.

* calculated from quantitative and censored values following a Bayesian approach (see report)

** estimated by calculating the 95% quantile of the marginal likelihood distribution (see report)

Analytical Methods

Element	Line Number	Method
Ca	2, 4, 5, 6, 8, 9, 10, 11	ICP-OES, dissolution with tartaric acid/HNO_3
	3	ICP-OES, dissolution with HNO_3/HF/HCl
	7	FAAS, dissolution with HNO_3, separation of SnO_2 and $Pb(NO_3)_2$
	12	ICP-OES, dissolution with HNO_3 and traces of tartaric acid
	13	ICP-OES, dissolution with HNO_3/fluoroboric acid
Sn	3, 4, 6, 8, 9, 11, 12	ICP-OES, dissolution with tartaric acid/HNO_3
	5	ICP-OES, dissolution with HNO_3 and traces of tartaric acid
	7	ICP-OES, dissolution with HNO_3/HF/HCl
	10	Gravimetry as SnO_2, dissolution with HNO_3
	13	ICP-OES, dissolution with HNO_3/fluoroboric acid
Bi	1, 2, 4, 5, 8, 9, 10	ICP-OES, dissolution with tartaric acid/HNO_3
	3	ICP-OES, dissolution with HNO_3 and traces of tartaric acid
	6	ICP-OES, dissolution with HNO_3/HF/HCl
	7	ICP-OES, dissolution with HNO_3/fluoroboric acid
Al	1, 2, 3, 4, 6, 7, 9, 10, 11	ICP-OES, dissolution with tartaric acid/HNO_3
	5	ICP-OES, dissolution with HNO_3/HF/HCl
	8	ICP-OES, dissolution with HNO_3 and traces of tartaric acid
	12	ICP-OES, dissolution with HNO_3/fluoroboric acid
Ag	1, 2, 3, 4, 7, 8, 9, 10, 11	ICP-OES, dissolution with tartaric acid/HNO_3
	5	ICP-MS, dissolution with HNO_3/HF/HCl
	6	FAAS, dissolution with HNO_3, separation of SnO_2 and $Pb(NO_3)_2$
Cu	1, 3, 4, 5, 6, 7, 8, 9, 11	ICP-OES, dissolution with tartaric acid/HNO_3
	2	FAAS, dissolution with HNO_3, separation of SnO_2 and $Pb(NO_3)_2$
	10	ICP-OES, dissolution with HNO_3/HF/HCl
	12	ICP-OES, dissolution with HNO_3 and traces of tartaric acid
Fe	2, 4, 5, 7, 8, 10, 11	ICP-OES, dissolution with tartaric acid/HNO_3
	3	Spectrophotometry, dissolution with HNO_3, separation of SnO_2
	6	ICP-MS, dissolution with HNO_3/HF/HCl
	9	ICP-OES, dissolution with HNO_3 and traces of tartaric acid
Sb	3	ICP-MS, dissolution with HNO_3/HF/HCl
	4, 5, 6, 8	ICP-OES, dissolution with tartaric acid/HNO_3
	7	ICP-OES, dissolution with HNO_3 and traces of tartaric acid
As	1, 3, 4, 7	ICP-OES, dissolution with tartaric acid/HNO_3
	2	ICP-MS, dissolution with HNO_3/HF/HCl
	5	ICP-OES, dissolution with HNO_3 and traces of tartaric acid
Cr	1, 3, 4, 5, 6, 8	ICP-OES, dissolution with tartaric acid/HNO_3
	2	ICP-MS, dissolution with HNO_3/HF/HCl
	7	ICP-OES, dissolution with HNO_3 and traces of tartaric acid
Mn	1, 2, 3, 4, 6, 8	ICP-OES, dissolution with tartaric acid/HNO_3
	5	ICP-MS, dissolution with HNO_3/HF/HCl
	7	ICP-OES, dissolution with HNO_3 and traces of tartaric acid
Se	1	ICP-MS, dissolution with HNO_3/HF/HCl
	2, 3, 4, 5, 6, 7	ICP-OES, dissolution with tartaric acid/HNO_3

Abbreviations:
FAAS – Flame atomic absorption spectrometry
ICP-OES – Inductively coupled plasma - optical emission spectrometry
ICP-MS – Inductively coupled plasma - mass spectrometry

Participating Laboratories

Aurubis AG, Hamburg, Germany
BAE Batterien GmbH, Berlin, Germany
Bundesanstalt für Materialforschung und -prüfung (BAM), Berlin, Germany
Nyrstar Stolberg, Stolberg, Germany
Clarios Germany GmbH & Co. KGaA, Hannover, Germany
Clarios, BTC Labs, Glendale WI, United States
Clarios Mexico, Monterrey Mexico
Clarios Zwickau GmbH & Co. KG, Zwickau, Germany
Ecobat Resources Freiberg GmbH, Freiberg, Germany
Hoppecke Batterien GmbH & Co. KG, Brilon-Hoppecke, Germany
Raghavendra Spectro Metallurgical Laboratory, Bangalore, India
TU Clausthal, Clausthal-Zellerfeld, Germany

Literature

A detailed technical report describing the analysis procedures and the treatment of the analytical data used to certify BAM-M113 is available on request or can be downloaded from BAM website (https://rrr.bam.de).

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Bundesanstalt für Materialforschung und -prüfung (BAM)

Dr. S. Richter
Committee for Certification

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Project Coordinator

BAM holds an accreditation as a reference material producer according to ISO 17034. This accreditation is valid only for the scope as specified in the certificate D-RM-11075-01-00. DAkkS is a signatory of the multilateral agreement (MLA) between EA, ILAC and IAF for mutual acceptance.	DAkkS Deutsche Akkreditierungsstelle D-RM-11075-01-00

This Certified Reference Material is offered by:

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