Bundesanstalt für Materialforschung und -prüfung (BAM)

in cooperation with the Committee of Chemists of the GDMB
Gesellschaft der Metallurgen und Bergleute e.V.

Certified Reference Material

BAM-M110

PbSb3

Certified Values

Element	Mass fraction [1] in %	Uncertainty [2] in %
As	0.107	0.008
Bi	0.0126	0.0004
Sb	3.08	0.08
Se	0.0106	0.0014
Sn	0.131	0.004
	in mg/kg	**in mg/kg**
Ag	22.6	1.7
Cu	6.4	0.4
Te	3.8	0.9

[1] Unweighted mean value of the means of accepted sets of data (consisting of at least 5 but usually 6 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).

This certificate is valid until 05/2048.

Sample description

The Reference Material is available in the form of discs (approx. 40 mm diameter and 30 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.

Values for information

Element	Mass fraction in mg/kg
Ca	< 2
Cd	< 1
Zn	< 1

Instructions for Use

Before use, the surface of the material must be prepared by milling or turning on a lathe. For wet chemical analysis chips have to be prepared by turning or milling of the sample surface.
An area of 8 mm in diameter in the centre of the discs should be avoided for spark optical emission spectrometry.

Transport and Storage

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

Metrological Traceability

The values are traceable to the SI (Système International d'Unités) via calibration using pure metals or substances of known stoichiometry or certified monoelement standard solutions. All values were confirmed in an inter-laboratory comparison using spark optical emission spectrometry.

Participating Laboratories

Aurubis AG, Hamburg, Germany
BERZELIUS Stolberg GmbH, Stolberg, Germany
Bundesanstalt für Materialforschung und -prüfung (BAM), Berlin, Germany
Hoppecke Batterien GmbH & Co. KG, Brilon-Hoppecke, Germany
Johnson Controls Sachsen-Batterien GmbH & Co. KG, Zwickau, Germany
Johnson Controls, VB Autobatterie GmbH & Co. KGaA, Hannover, Germany
Muldenhütten Recycling und Umwelttechnik GmbH, Freiberg, Germany
WESER METALL GmbH, Nordenham, Germany

Technical Report

A detailed technical report describing the analysis procedures and the treatment of the analytical data used to certify BAM-M110 is available on request or can be downloaded from BAM website (www.bam.de).

Means of Accepted Data Sets

Certified values — values for information

mass fraction in % — mass fraction in mg/kg — mass fraction in mg/kg

Line No.	As	Bi	Sb	Se	Sn		Ag	Cu	Te		*Ca*	*Cd*	*Zn*
1	0.096	0.0120	2.965	0.0091	0.1268		20.4	6.01	2.62		*0.97*	*0.13*	*0.27*
2	0.102	0.0123	2.970	0.0092	0.1277		21.1	6.09	3.52		*< 1*	*0.21*	*0.29*
3	0.103	0.0123	3.012	0.0095	0.1284		21.3	6.12	3.60		*< 1*	*< 1*	*< 1*
4	0.106	0.0124	3.031	0.0105	0.1287		22.3	6.32	3.56		*< 1*	*< 1*	*< 1*
5	0.107	0.0126	3.047	0.0111	0.1288		22.4	6.39	3.62		*1.80*	*< 1*	*< 1*
6	0.109	0.0126	3.058	0.0112	0.1289		22.6	6.66	4.14		---	*< 1*	*< 1*
7	0.110	0.0128	3.108	0.0116	0.1355		25.2	6.90	5.34			*< 1*	*< 1*
8	0.110	0.0130	3.250	0.0121	0.1359		25.3	---				---	
9	0.114	0.0131	3.257		0.1397								
10	0.117												
11													
12													
13													
M	0.107	0.0126	3.078	0.0106	0.1311		22.6	6.36	3.77		*< 2*	*< 1*	*< 1*
s_M	0.007	0.0004	0.109	0.0012	0.0046		1.9	0.33	0.83				
$\overline{s}_i$	0.003	0.0003	0.040	0.0005	0.0014		0.5	0.18	0.20				

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 (Grubbs 95 %). A data set consists of at least 6 single values of one laboratory.

M : mean of laboratory means

s_M : standard deviation of laboratory means

$\overline{s}_i$: averaged repeatability standard deviation (square root of the mean of laboratory variances)

Note: "< - values" were not included into the calculation of M and s_M

Analytical Methods used for Certification

Element	Line Number	Method
As	1	ICP-OES, dissolution with $HNO_3/HClO_4$
	2	ICP-OES, dissolution with tartaric acid/HNO_3/HF
	3, 4, 8, 10	ICP-OES, dissolution with tartaric acid/HNO_3
	5	FAAS, dissolution with HNO_3/HF
	6	ICP-OES, dissolution with tartaric acid/HNO_3, separation of Pb as sulfate
	7	ICP-OES, dissolution with HNO_3/HF
	9	ICP-OES, dissolution with HNO_3/HCl, separation of Pb as chloride
Bi	1, 2, 6, 7	ICP-OES, dissolution with tartaric acid/HNO_3
	3	FAAS, dissolution with HNO_3/HCl, separation of Pb as chloride
	4	ICP-OES, dissolution with tartaric acid/HNO_3, separation of Pb as sulfate
	5	ICP-OES, dissolution with HNO_3/HCl, separation of Pb as chloride
	8	ICP-OES, dissolution with HNO_3/HF
	9	ICP-OES, dissolution with tartaric acid/HNO_3/HF/H_2O_2
Sb	1, 2, 3, 4, 5, 7	ICP-OES, dissolution with tartaric acid/HNO_3
	6	ICP-OES, dissolution with HNO_3/HF
	8	Titration with bromate, dissolution with HCl/HF/H_2SO_4
	9	FAAS, dissolution with HNO_3/HCl, separation of Pb as chloride
Sn	1	ICP-OES, dissolution with tartaric acid/HNO_3, separation of Pb as sulfate
	2, 5, 7, 8	ICP-OES, dissolution with tartaric acid/HNO_3
	3	ICP-OES, dissolution with tartaric acid/HNO_3/HF/H_2O_2
	4	ICP-OES, dissolution with HNO_3/HF
	6	FAAS, dissolution with HNO_3/HF
	9	ICP-OES, dissolution with HNO_3/HCl, separation of Pb as chloride
Se	1, 2, 7	ICP-OES, dissolution with tartaric acid/HNO_3
	3	ICP-OES, dissolution with HNO_3/HF
	4	ICP-OES, dissolution with HNO_3
	5	ICP-OES, dissolution with tartaric acid/HNO_3, separation of Pb as sulfate
	6	ICP-OES, dissolution with tartaric acid/HNO_3/HF/H_2O_2
	8	ICP-OES, dissolution with HNO_3/HCl, separation of Pb as chloride
Ag	1, 2, 4, 5	ICP-OES, dissolution with tartaric acid/HNO_3
	3	ICP-OES, dissolution with HNO_3/HF
	6	ICP-OES, dissolution with tartaric acid/HNO_3/HF/H_2O_2
	7	ICP-OES, dissolution with HNO_3/HCl, separation of Pb as chloride
	8	ICP-OES, dissolution with tartaric acid/HNO_3, separation of Pb as sulfate

Element	Line Number	Method
Cu	1, 2, 4	ICP-OES, dissolution with tartaric acid/HNO_3
	3	ICP-OES, dissolution with HNO_3/HF
	5	ICP-OES, dissolution with tartaric acid/HNO_3/HF/H_2O_2
	6	ICP-OES, dissolution with tartaric acid/HNO_3, separation of Pb as sulfate
	7	ETAAS, dissolution with HNO_3/HF
Te	1, 4, 5	ICP-OES, dissolution with HNO_3/HF
	2	ICP-OES, dissolution with tartaric acid/HNO_3
	3	ETAAS, dissolution with HNO_3/HF
	6	ICP-OES, dissolution with tartaric acid/HNO_3/HF/H_2O_2
	7	ICP-OES, dissolution with tartaric acid/HNO_3, separation of Pb as sulfate
Ca	*1, 5*	*ICP-OES, dissolution with tartaric acid/HNO_3*
	2	*ICP-OES, dissolution with tartaric acid/HNO_3, separation of Pb as sulfate*
	3	*ICP-OES, dissolution with tartaric acid/HNO_3/HF/H_2O_2*
	4	*ICP-OES, dissolution with HNO_3/HF*
Cd	*1, 2, 6, 7*	*ICP-OES, dissolution with tartaric acid/HNO_3*
	3	*ICP-OES, dissolution with tartaric acid/HNO_3, separation of Pb as sulfate*
	4	*ICP-OES, dissolution with tartaric acid/HNO_3/HF/H_2O_2*
	5	*ICP-OES, dissolution with HNO_3/HF*
Zn	*1, 2, 6, 7*	*ICP-OES, dissolution with tartaric acid/HNO_3*
	3	*ICP-OES, dissolution with tartaric acid/HNO_3, separation of Pb as sulfate*
	4	*ICP-OES, dissolution with tartaric acid/HNO_3/HF/H_2O_2*
	5	*ICP-OES, dissolution with HNO_3/HF*

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

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Bundesanstalt für Materialforschung und -prüfung (BAM)

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Inorganic Reference Materials

BAM holds an accreditation as a reference material producer according to ISO Guide 34 in combination with ISO/IEC 17025. 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

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