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-M109

Refined Lead

Certified Values

Element	Mass fraction [1] in %	Uncertainty [2] in %
As	0.0113	0.0006
Bi	0.0193	0.0006
Sb	0.0098	0.0003
Sn	0.115	0.004
	in mg/kg	**in mg/kg**
Ag	45.1	1.0
Cd	35.3	0.9
Cu	19.6	0.7
Ni	3.5	0.3
Te	30.6	1.5
Tl	3.0	0.5
Zn	31.8	2.1

[1] Unweighted mean value of the means of accepted sets of data, each set being obtained by at least 4 laboratories and/or with different methods of measurement. The values are traceable to the SI (Système International d'Unités) by the use of pure substances of known stoichiometry for calibration.

[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 03/2048.

Sample description

The Reference Material is available in the form of discs (approx. 40 mm diameter and 40 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 0.1 g.

Values for information

Element	Mass fraction in mg/kg
Al	< 2.1
In	< 0.5

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 8mm 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. All values were confirmed in an inter-laboratory comparison using spark optical emission spectrometry.

Participating Laboratories

Aurubis AG, Hamburg, Germany
Bundesanstalt für Materialforschung und -prüfung (BAM), Berlin, Germany
Berzelius Stolberg, Stolberg, 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
Recyclex Harz-Metall GmbH, Goslar, Germany
TAB-IPM d.o.o., Žerjav, Slovenia
ThyssenKrupp Steel Europe, Duisburg, 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-M109 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**	**Sn**		**Ag**	**Cd**	**Cu**	**Ni**	**Te**	**Tl**	**Zn**		***Al***	***In***
1	0.0107	0.0176	0.0093	0.106		43.9	33.4	18.5	2.97	---	2.12	29.5		*0.61*	*0.3*
2	0.0107	0.0182	0.0094	0.110		44.5	34.5	18.6	3.00	28.6	2.51	30.5		*1.42*	
3	0.0108	0.0185	0.0095	0.112		44.7	34.7	18.8	3.02	29.4	2.53	31.4		*2.08*	
4	0.0110	0.0190	0.0096	0.113		44.7	35.1	18.8	3.30	29.7	3.00	31.5			
5	0.0110	0.0191	0.0097	0.114		44.9	35.1	18.9	3.31	29.7	3.22	31.6			
6	0.0112	0.0193	0.0097	0.114		45.0	35.2	19.4	3.33	30.4	3.47	31.8			
7	0.0117	0.0197	0.0098	0.114		45.1	35.4	19.9	3.36	30.4	3.50	32.4			
8	0.0117	0.0199	0.0100	0.116		46.1	35.6	19.9	3.49	30.5	3.86	32.6			
9	0.0118	0.0200	0.0101	0.117		46.7	35.6	20.0	3.81	30.8		32.7			
10	0.0119	0.0204	0.0103	0.119			35.7	20.3	3.90	31.3		32.8			
11	0.0122	0.0207	0.0104	0.119			36.2	20.9	4.01	32.2		33.5			
12				0.125			36.8	21.0	4.12	33.7		---			
M	0.0113	0.0193	0.0098	0.115		45.1	35.3	19.6	3.47	30.6	3.03	31.8		*1.37*	
s_M	0.0006	0.0010	0.0004	0.005		0.9	0.9	0.9	0.40	1.5	0.60	1.2		*0.74*	
$\overline{s}_\mathrm{i}$	0.0003	0.0004	0.0003	0.002		0.5	0.8	0.4	0.27	0.7	0.28	1.1		*0.46*	

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 4 but usually 6 single values of one laboratory.

M : mean of laboratory means

s_M : standard deviation of laboratory means

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

Analytical Method used for Certification

Element	Line Number	Method
As	1, 3, 4, 6, 7, 8, 9, 10, 11	ICP-OES, dissolution with tartaric acid/HNO_3
	2	ICP-OES, dissolution with HNO_3/tetraborate
	5	ICP-OES, dissolution with HNO_3/HF
Bi	1, 2, 3, 4, 5, 6, 8, 9, 10	ICP-OES, dissolution with tartaric acid/HNO_3
	7	ICP-OES, dissolution with HNO_3/HF
	11	ICP-OES, dissolution with HNO_3/tetraborate
Sb	1, 4, 5, 6, 7, 8, 9, 10, 11	ICP-OES, dissolution with tartaric acid/HNO_3
	2	ICP-OES, dissolution with HNO_3/tetraborate
	3	ICP-OES, dissolution with HNO_3/HF
Sn	1, 2, 3, 4, 8, 9, 10, 11, 12	ICP-OES, dissolution with tartaric acid/HNO_3
	5	ICP-OES, dissolution with HNO_3/tetraborate
	6	FAAS, dissolution with HNO_3/HF
	7	ICP-OES, dissolution with HNO_3/HF
Ag	1, 3, 4, 5, 7	ICP-OES, dissolution with tartaric acid/HNO_3
	2, 9	FAAS, dissolution with tartaric acid/HNO_3
	6	ICP-OES, dissolution with HNO_3/HF
	8	ICP-OES, dissolution with HNO_3/tetraborate
Cd	1, 2, 3, 5, 6, 8, 9, 10, 11	ICP-OES, dissolution with tartaric acid/HNO_3
	4	ICP-OES, dissolution with HNO_3/HF
	7	ICP-OES, dissolution with HNO_3/tetraborate
	10	ETAAS, dissolution with HNO_3/HF
Cu	1, 2, 3, 4, 5, 6, 8, 9, 12	ICP-OES, dissolution with tartaric acid/HNO_3
	7	ETAAS, dissolution with HNO_3/HF
	10	ICP-OES, dissolution with HNO_3/HF
	11	ICP-OES, dissolution with HNO_3/tetraborate
Ni	1, 2, 3, 4, 5, 6, 7, 8, 12	ICP-OES, dissolution with tartaric acid/HNO_3
	9	ETAAS, dissolution with HNO_3/HF
	10	ICP-OES, dissolution with HNO_3/HF
	11	ICP-OES, dissolution with HNO_3/tetraborate
Te	2	ICP-OES, dissolution with HNO_3/tetraborate
	3	ICP-OES, dissolution with HNO_3/HF
	4, 5, 6, 7, 8, 9, 10, 11	ICP-OES, dissolution with tartaric acid/HNO_3
	12	ETAAS, dissolution with HNO_3/HF
Tl	1, 2, 3, 4, 8	ICP-OES, dissolution with tartaric acid/HNO_3
	5	ICP-OES, dissolution with HNO_3/HF
	6	ETAAS, dissolution with HNO_3/HF
	7	ICP-MS, dissolution with HNO_3/tetraborate
Zn	1, 2, 3, 4, 6, 8, 9, 10	ICP-OES, dissolution with tartaric acid/HNO_3
	5	ICP-OES, dissolution with HNO_3/HF
	7	FAAS, dissolution with HNO_3/HF
	11	ICP-OES, dissolution with HNO_3/tetraborate

Element	Line Number	Method
Al	*1*	*ICP-OES, dissolution with HNO_3/tetraborate*
	2, 3	*ICP-OES, dissolution with tartaric acid/HNO_3*
In	*1*	*ICP-OES, dissolution with HNO_3/tetraborate*

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

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Analytical Chemistry;
Reference Materials

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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-10975-01-00. 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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