in cooperation with the WG 'Lead'
of the Committee of Chemists of GDMB

European Reference Materials

CERTIFICATE OF ANALYSIS

ERM®-EB103

PbSb1,6			
	Certified value [1]	Uncertainty [2]	
Element	Mass fraction in %		
Sb	1.64	±	0.06
As	0.097	±	0.004
Sn	0.183	±	0.026
Element	Mass fraction in mg/kg		
Se	180	±	10
Bi	158	±	4
Ag	66	±	6
Cu	9.7	±	0.9
Tl	15.2	±	0.7
Ni	3.02	±	0.27
Cd	0.20	±	0.08

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

[2] Estimated expanded uncertainty *U* with a coverage factor of about *k*=2, corresponding to a level of confidence of about 95 %, as defined in the Guide to the Expression of Uncertainty in Measurement (1995) ISO, Geneva.

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This certificate is valid until 09/2056; this validity may be extended as further evidence of stability becomes available.
The minimum sample size for wet chemical analysis is 1 g.

NOTE

European Reference Material ERM®-EB103 was produced and certified under the responsibility of Bundesanstalt für Materialforschung und –prüfung (BAM) in cooperation with the Committee of Chemists of the GDMB, Gesellschaft für Bergbau, Metallurgie, Rohstoff- und Umwelttechnik according to the principles laid down in the technical guidelines of the European Reference Materials® co-operation agreement between BAM-LGC-IRMM. Information on these guidelines is available on the Internet (http://www.erm-crm.org).

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Indicative Values[1)]			
	Indicative value [2)]	Uncertainty [3)]	
Element	Mass fraction in mg/kg		
S	5.4	±	1.2
Te	1.9	±	0.6

[1)] Values were not certified, but given as indicative values because the number of accepted data sets was considered to be too low, when the spread from the round robin certification was considerably larger than the state of the practice or when only 'less than' values were reported from the round robin certification.

[2)] Unweighted mean value of the means of accepted sets of data, each set being obtained in a different laboratory and/or with a different method of measurement. The values are traceable to the SI (Système International d'Unités) by the use of sufficiently pure substances of known stoichiometry for calibration.

[3)] Estimated expanded uncertainty *U* with a coverage factor of about *k*=2, corresponding to a level of confidence of about 95 %, as defined in the Guide to the Expression of Uncertainty in Measurement (1995) ISO, Geneva.

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DESCRIPTION OF THE SAMPLE

The Reference Material is available in the form of discs (40 mm diameter, 30 mm thickness).

MEANS OF ACCEPTED DATA SETS (FOR ONE METHOD AT ONE LABORATORY, RESPECTIVELY)

Certified values
Mass fraction in %

Mass fraction in mg/kg

Indicative values
Mass fraction in mg/kg

Line No.	Sb	As	Sn	Se	Bi	Ag	Cu	Tl	Ni	Cd	*S*	*Te*
1	1.597	0.0918	0.1791	157.2	153.0	62.29	8.08	13.86	2.67	0.137	*4.22*	*1.63*
2	1.603	0.0919	0.1795	164.8	154.4	62.46	8.67	13.96	2.77	0.141	*4.64*	*1.65*
3	1.616	0.0925	0.1801	169.7	156.0	63.00	9.32	15.03	2.83	0.167	*5.49*	*1.87*
4	1.627	0.0947	0.1803	178.4	157.3	64.42	9.44	15.08	2.86	0.210	*6.21*	*2.50*
5	1.630	0.0960	0.1805	179.6	157.7	64.50	9.44	15.45	2.96	0.232	*6.54*	
6	1.634	0.0960	0.1836	181.1	158.1	66.00	9.51	15.63	3.03	0.239		
7	1.643	0.0977	0.1838	184.7	158.5	67.12	9.66	15.70	3.06	0.273		
8	1.654	0.0977	0.1842	185.0	158.8	67.28	9.67	16.05	3.07			
9	1.655	0.0987	0.1864	192.3	164.2	67.59	10.13	16.17	3.45			
10	1.655	0.0998	0.1868		164.4	67.65	10.92		3.48			
11	1.664	0.0998	0.1898			68.04	11.48					
12	1.670	0.1026	---			71.49						
13	1.707											
M :	1.643	0.0966	0.1831	177.0	158.23	65.99	9.67	15.21	3.02	0.200	*5.42*	*1.91*
s_M:	0.030	0.0034	0.0035	11.06	3.68	2.738	0.94	0.83	0.27	0.053	*1.00*	*0.41*
$\overline{s_i}$:	0.027	0.0029	0.0023	5.77	3.18	1.810	0.49	0.95	0.21	0.023	*1.305*	*0.40*

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 99%).

M: mean of means of data sets
s_M: standard deviation of means of data sets*
$\overline{s_i}$: mean of standard deviations of data sets

*calculated of at least 3 but usually 6 single values

ANALYTICAL METHOD USED FOR CERTIFICATION

Element	Line no.	Method
Sb	1, 4, 5, 6, 7, 8, 10, 11, 13	Inductively coupled plasma – optical emission spectrometry (ICP OES)
	2, 3, 12	Flame atomic absorption spectrometry (F AAS)
	9	Titration
As	1, 2, 4, 5, 6, 7, 9, 11	Inductively coupled plasma – optical emission spectrometry (ICP OES)
	3, 8, 10	Flame atomic absorption spectrometry (F AAS)
	12	Electrothermal atomic absorption spectrometry (ET AAS)
Sn	1, 4, 5, 6, 7, 9, 10, 11	Inductively coupled plasma – optical emission spectrometry (ICP OES)
	2, 3, 8	Flame atomic absorption spectrometry (F AAS)
Se	1	Hydride generation - atomic absorption spectrometry (H-AAS)
	2, 3, 5, 8, 9	Inductively coupled plasma – optical emission spectrometry (ICP OES)
	4	Flame atomic absorption spectrometry (F AAS)
	6	Electrothermal atomic absorption spectrometry (ET AAS)
	7	Atomic fluorescence spectrometry
Bi	1, 2, 5, 7, 9, 10	Inductively coupled plasma – optical emission spectrometry (ICP OES)
	3, 4, 6, 8	Flame atomic absorption spectrometry (F AAS)
Ag	1, 3, 4, 10	Flame atomic absorption spectrometry (F AAS)
	2, 5, 6, 7, 8, 9, 11, 12	Inductively coupled plasma – optical emission spectrometry (ICP OES)
Cu	1, 2, 4, 5, 6, 7, 9	Inductively coupled plasma – optical emission spectrometry (ICP OES)
	3, 8, 10, 11	Flame atomic absorption spectrometry (F AAS)
Tl	1, 2, 3, 5, 6, 9	Inductively coupled plasma – optical emission spectrometry (ICP OES)
	4, 7	Flame atomic absorption spectrometry (F AAS)
	8	Electrothermal atomic absorption spectrometry (ET AAS)
Ni	1, 2, 3, 4, 6, 8, 10	Inductively coupled plasma – optical emission spectrometry (ICP OES)
	5, 7	Electrothermal atomic absorption spectrometry (ET AAS)
	9	Flame atomic absorption spectrometry (F AAS)
Cd	1, 2, 5, 6	Inductively coupled plasma – optical emission spectrometry (ICP OES)
	3	Flame atomic absorption spectrometry (F AAS)
	4, 7	Electrothermal atomic absorption spectrometry (ET AAS)
S	*1, 3, 5*	*Inductively coupled plasma – optical emission spectrometry (ICP OES)*
	2, 4	*Spectrophotometry*
Te	*1, 2, 4*	*Inductively coupled plasma – optical emission spectrometry (ICP OES)*
	3	*Flame atomic absorption spectrometry (F AAS)*

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PARTICIPANTS

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- BSB Recycling GmbH, Braubach (Germany)
- Bundesanstalt für Materialforschung und -prüfung (BAM), Working group „Metal Analysis; Metal Reference Materials“, Berlin (Germany)
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- Metaleurop Weser GmbH, Nordenham (Germany)
- Metalllabor Duisburg, Duisburg (Germany)
- Muldenhütten Recycling und Umwelttechnik GmbH, Freiberg (Germany)
- Norddeutsche Affinerie AG, Hamburg (Germany)
- Steco Batteries, Outarville (France)
- TU Bergakademie Freiberg, Freiberg (Germany)
- Umicore Hoboken, Hoboken (Belgium)
- Umicore Overpelt, Overpelt (Belgium)
- VB Autobatterie GmbH, Hannover (Germany)

INTENDED USE

The CRM is intended for establishing and checking the calibration of optical emission and X-ray spectrometers for the analysis of samples of similar materials.

INSTRUCTIONS FOR USE

Before use, the surface of the material must be cleaned by milling or turning on a lathe. An area of approx. 10 mm diameter in the centre of the discs should not be used for spark emission spectrometry.

STORAGE

The material should be stored at ambient conditions in a dry and clean environment.

TECHNICAL REPORT

A detailed technical report (in German) describing the analysis procedures and the treatment of the analytical data used to certify ERM®-EB103 is available on request.

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Supply of Reference Materials by Bundesanstalt für Materialforschung und –prüfung:

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