



CERTIFICATE OF ANALYSIS

ERM[®]-EB102a

PbCaSn

| | Certified Value | S | | | | |
|------------------------------|--|---------------|------------------------------|--|--|--|
| | Certified value 1) | | Uncertainty ²⁾ | | | |
| Element | Mass | fraction in 9 | % | | | |
| Са | 0.0635 | | 0.0022 | | | |
| Sn | 1.01 | ± | 0.05 | | | |
| Element | Mass fraction in mg/kg | | | | | |
| AI | 124 | ± | 11 | | | |
| Ag | 170 | ± | 6 | | | |
| Bi | 73.7 | ± | 2.6 | | | |
| Cu | 1.3 | ± | 0.4 | | | |
| TI | 30.2 | ± | 1.5 | | | |
| laboratory and/or with a dif | f the means of accepted sets of da ferent method of measurement. Th alibration using pure metals or subs | e values are | traceable to the SI (Système | | | |

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, ISO, 1993.

This certificate is valid until 10/2059; this validity may be extended as further evidence of stability becomes available.

DESCRIPTION OF THE SAMPLE

The Reference Material is available in the form of discs (40 mm diameter and 40 mm height). It is intended for establishing and checking the calibration of optical emission and X-ray spectrometers (excluding microanalysis) for the analysis of samples of similar materials.

NOTE

European Reference Material ERM[®]-EB102a was produced and certified under the responsibility of BAM Federal Institute for Materials Research and Testing 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 (<u>http://www.erm-crm.org</u>).



Accepted as an ERM[®], Berlin, 2009-09-30.

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| Indicative Values ¹⁾ | | | | | | |
|---|------------------------|----------------|--|--|--|--|
| | Indicative value 2) | Uncertainty 3) | | | | |
| Element | Mass fraction in mg/kg | | | | | |
| Sb | 4 | ± 4 | | | | |
| As | < 2 | | | | | |
| In | < 2 | | | | | |
| Те | < 1.1 | | | | | |
| Zn | < 0.5 | | | | | |
| ¹⁾ Values were not certified, but given as indicative values, when the number of accepted data sets was considered to be too low (< 5), when the uncertainty from the inter-laboratory certification was considerably larger than the expected range or when only an upper limit can be given. | | | | | | |
| 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) via calibration using pure substances of known stoichiometry. | | | | | | |

³⁾ 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, ISO, 1993.

| Additional Material Information ¹⁾ | | | | | |
|---|--------------------------------------|--------------------------|--|--|--|
| | Mean or upper limit | Stddev ²⁾ | | | |
| Element | Mass fraction in mg/kg | | | | |
| Fe | < 2 | | | | |
| Mg | < 1 | | | | |
| Na | 4 | ± 1 | | | |
| S | < 3 | | | | |
| ¹⁾ Values were obtained in an | interlaboratory comparison with spar | k emission spectrometry. | | | |

²⁾ Standard deviation calculated from the mean of means of the interlaboratory comparison with n = 6 for Fe, n = 4 for Mg, n = 8 for Na and n = 12 for S.



MEANS OF ACCEPTED DATA SETS

| | d values action in | - | Mass fra | iction in I | mg/kg | | | - | ndicati Mass fra | | | | |
|------------------|-----------------------|-------|----------|-------------|-------|------|------|---|----------------------------|-------|-------|-------|--------|
| _ine no. | Ca | Sn | AI | Ag | Bi | Cu | ТІ | | Sb | As | In | Те | Zn |
| 1 | 0.0582 | | 111 | 165 | 67.0 | 0.56 | 28.7 | | 0.62 | 0.11 | 0.15 | 0.08 | 0.38 |
| 2 | 0.0613 | 0.937 | 119 | 166 | 70.2 | 0.62 | 29.0 | | 0.94 | 0.63 | 0.32 | 0.43 | 0.41 |
| 3 | 0.0616 | 0.962 | 119 | 167 | 70.7 | 0.84 | 29.1 | | 1.03 | 0.81 | 1.00 | 0.50 | 0.45 |
| 4 | 0.0617 | 0.999 | 120 | 168 | 72.1 | 0.98 | 29.1 | | 2.13 | 2.00 | 1.44 | 1.05 | 0.47 |
| 5 | 0.0622 | 1.011 | 121 | 168 | 72.2 | 1.00 | 29.6 | | 5.08 | < 0.1 | < 1 | < 1.5 | 0.53 |
| 6 | 0.0632 | 1.013 | 122 | 169 | 72.2 | 1.03 | 29.6 | | 6.87 | < 0.4 | < 3 | < 1 | 0.55 |
| 7 | 0.0633 | 1.016 | 126 | 171 | 74.7 | 1.20 | 31.1 | | 8.87 | < 1 | | < 1 | |
| 8 | 0.0654 | 1.023 | 126 | 172 | 75.7 | 1.21 | 32.0 | | < 1 | | | | < 0.5 |
| 9 | 0.0662 | 1.028 | 127 | 172 | 76.1 | 1.45 | 33.3 | | < 2.5 | | | | < 1 |
| 10 | 0.0671 | 1.031 | 131 | 172 | 76.1 | 1.45 | | | | | | | |
| 11 | 0.0680 | 1.039 | 131 | 175 | 77.4 | 1.71 | | | | | | | |
| 12 | | 1.040 | 133 | 178 | 79.7 | 1.79 | | | | | | | |
| 13 | | 1.053 | | | | 2.08 | | | | | | | |
| 14 | | | | | | 2.09 | | | | | | | |
| М: | 0.0635 | 1.01 | 124 | 170 | 74 | 1.3 | 30.2 | | 3.5 | 0.886 | 0.726 | 0.51 | 0.947 |
| S _M : | 0.0030 | 0.04 | 7 | 4 | 4 | 0.5 | 1.6 | | 3.3 | 0.799 | 0.602 | 0.401 | 0.2882 |
| S _i : | 0.0008 | 0.008 | 2 | 2 | 1.21 | 0.19 | 1.1 | | 0.23 | 0.075 | 0.055 | 0.101 | 0.128 |

The laboratory mean values have been examined statistically to eliminate outlying values. Each laboratory mean consists of at least 4 but usually 6 single values. Where "---" appears in the table it indicates that an outlying value has been omitted (Grubbs 99 %). " < "-values have not been considered in statistical evaluation.

M : mean of means of data sets

 \overline{s}_i : mean of standard deviations of data sets under repeatability conditions

 s_M : standard deviation of means of data sets

TECHNICAL REPORT

A detailed technical report (in German) describing the analysis procedures and the treatment of the analytical data used to certify ERM[®]-EB102a is available on request.



ANALYTICAL METHOD USED FOR CERTIFICATION

| Element | Line no. | Method | | | |
|----------------|---|--|--|--|--|
| Ca | 1, 8, 9 2, 3, 4, 5, 6, 7, 10 ,11 | FAAS ICP-OES | | | |
| Sn | 2, 4, 5, 6, 7, 8, 9, 10, 11, 13 3, 12 | ICP-OES FAAS | | | |
| AI | 1, 2, 4, 5, 6, 7, 9, 11, 12 3, 10 8 | ICP-OES FAAS FAAS after separation of the matrix | | | |
| Ag | 1 2, 3, 5, 6, 7, 8, 9, 11, 13 4 10, 11, 12 | FAAS after separation of the matrix ICP-OES ICP-MS FAAS | | | |
| Bi | 1, 2, 3, 6, 8, 9, 10, 11, 12 4, 5 7 | ICP-OES FAAS ICP-MS | | | |
| TI | 1, 2, 7, 8, 9 3, 4 5, 6 | ICP-OES FAAS ICP-MS | | | |
| Cu | 1 2, 3, 5, 6, 8, 10, 11, 12 4, 9 7, 13 14 | ICP-OES after separation of the matrix ICP-OES ICP-MS FAAS ETAAS | | | |
| Sb | 1, 8 2, 3, 4, 5, 6, 9 7 | ICP-MS ICP-OES FAAS | | | |
| As | 1 2, 3, 6 4 5, 7 | ICP-OES after separation of the matrix ICP-OES Spectrophotometry ICP-MS | | | |
| In | 1 2, 3, 4, 6 5 | ICP-OES after separation of the matrix ICP-OES ICP-MS | | | |
| Te | 1 2, 3, 4, 5 6, 7 | ICP-OES after separation of the matrix ICP-OES ICP-MS | | | |
| Zn | 1, 2, 3, 4 5 6 8, 9 | ICP-OES ICP-MS ICP-OES after separation of the matrix FAAS | | | |
| Abbreviations: | | | | | |

Abbreviations:

| ductively coupled plasma optical emission spectrometry |
|--|
| ductively coupled plasma mass spectrometry |
| ame atomic absorption spectrometry |
| ectrothermal atomic absorption spectrometry |
| |



INTENDED USE

The CRM is intended for establishing and checking the calibration of optical emission and X-ray spectrometers (excluding micro-analysis) for the analysis of samples of similar materials. The minimum sample size for wet chemical analysis is 0.5 g.

INSTRUCTIONS FOR USE

Before use, the surface of the material must be prepared by milling or turning on a lathe.

STORAGE

The material should be stored in a dry and clean environment at room temperature.

PARTICIPANTS

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Supply of Reference Materials by: BAM Bundesanstalt für Materialforschung und -prüfung

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