

CERTIFICATE OF ANALYSIS

ERM[®]-EB374

CuSn8

| Certified Values | | |
|------------------|-------------------------------|---------------------------|
| Element | Certified value ¹⁾ | Uncertainty ²⁾ |
| | Mass fraction in % | |
| Cu | 92.22 | ± 0.05 |
| Sn | 7.60 | ± 0.13 |
| P | 0.170 | ± 0.008 |
| | Mass fraction in mg/kg | |
| Ag | 12.1 | ± 1.3 |
| Fe | 40 | ± 4 |
| Mn | 4.3 | ± 0.3 |
| Ni | 32.7 | ± 1.3 |
| Pb | 8.3 | ± 0.9 |
| Zn | 40.4 | ± 1.9 |

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

²⁾ Estimated expanded uncertainty U with a coverage factor of about $k=2$, corresponding to a level of confidence of 95 %, as defined in the Guide to the expression of uncertainty in measurement, ISO, 1993. For Sn and P inhomogeneity contributed significantly to the uncertainty.

This certificate is valid until 09/2053; this validity may be extended as further evidence of stability becomes available.

The minimum sample size for wet chemical analysis is 0.5 g.

NOTE

European Reference Material ERM[®]-EB374 was originally certified as BAM-374. It 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 ³⁾ | | |
|--|--------------------------------|---------------------------|
| | Indicative value ⁴⁾ | Uncertainty ⁵⁾ |
| Element | Mass fraction in mg/kg | |
| As | 4.3 | ± 1.2 |
| Bi | 2.2 | ± 1.3 |
| S | 13 | ± 5 |
| Sb | 6.3 | ± 1.4 |
| Element | Mass fraction in mg/kg | |
| Al, Cd, Co, Cr, Mg, Te, Ti, Zr | < 1 | |
| Se | < 2 | |
| Si | < 10 | |
| <p>³⁾ Values were not certified, but given as indicative values, when 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 'lower as' values were reported from the round robin certification.</p> <p>⁴⁾ Unweighted mean value of the means of accepted sets of data, each set being obtained in a different laboratory and/or a different method of measurement. The values are traceable to the SI (Système International d'Unités) via calibration using sufficiently pure substances of known stoichiometry.</p> <p>⁵⁾ Estimated expanded uncertainty U with a coverage factor of about $k=2$, corresponding to a level of confidence of 95 %, as defined in the Guide to the expression of uncertainty in measurement, ISO, 1993.</p> | | |

DESCRIPTION OF THE SAMPLE

The Reference Material is available in the form of discs (40 mm diameter and 30 mm height). It is intended for establishing and checking the calibration of optical emission and X-ray spectrometers for the analysis of samples of similar materials

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

| Line No. | Mass fraction in % | | | Mass fraction in mg/kg | | | | | | | | | |
|------------------------|--------------------|------|--------|------------------------|-----|------|------|--------|------|--------|-----|-------|-----|
| | Cu | Sn | P | Ag | Fe | Mn | Ni | Pb | Zn | As | Bi | S | Sb |
| 1 | 92.15 | 7.50 | - | 9.3 | 31 | 3.4 | 27.0 | 6.6 | 35.9 | 3.2 | 1.7 | (< 5) | 4.3 |
| 2 | 92.17 | 7.51 | 0.1665 | 11.1 | 33 | 3.8 | 28.7 | 7.8 | 36.2 | 3.5 | 2.3 | 9.12 | 5.2 |
| 3 | 92.18 | 7.53 | 0.1666 | 11.8 | 34 | 4.1 | 30.2 | 8.0 | 36.5 | 3.7 | 2.7 | 12.5 | 6.3 |
| 4 | 92.19 | 7.57 | 0.1693 | 12.0 | 35 | 4.3 | 31.8 | 8.0 | 37.4 | 4.3 | | 14.1 | 6.6 |
| 5 | 92.22 | 7.57 | 0.1697 | 12.2 | 39 | 4.4 | 31.9 | 8.3 | 38.4 | 5.3 | | 15.2 | 7.6 |
| 6 | 92.25 | 7.59 | 0.1703 | 12.3 | 40 | 4.4 | 32.7 | 8.3 | 38.7 | 5.9 | | | 7.7 |
| 7 | 92.25 | 7.60 | 0.1724 | 12.4 | 40 | 4.5 | 33.0 | 9.7 | 38.7 | (< 10) | | | |
| 8 | 92.29 | 7.60 | 0.1730 | 12.6 | 42 | 4.5 | 33.1 | 10.0 | 39.5 | | | | |
| 9 | 92.31 | 7.65 | | 15.3 | 43 | 4.6 | 33.2 | - | 40.5 | | | | |
| 10 | | 7.69 | | - | 43 | 4.6 | 33.3 | (< 10) | 40.8 | | | | |
| 11 | | 7.76 | | | 44 | 4.8 | 33.3 | (< 5) | 41.0 | | | | |
| 12 | | | | | 46 | 4.9 | 33.8 | | 41.0 | | | | |
| 13 | | | | | 48 | - | 34.5 | | 44.4 | | | | |
| 14 | | | | | 50 | | 35.4 | | 45.7 | | | | |
| 15 | | | | | | | 35.7 | | 45.8 | | | | |
| 16 | | | | | | | 35.7 | | 46.3 | | | | |
| <i>M</i> : | 92.22 | 7.60 | 0.1697 | 12.1 | 40 | 4.3 | 32.7 | 8.3 | 40.4 | 4.3 | 2.2 | 12.7 | 6.3 |
| <i>s_M</i> : | 0.05 | 0.08 | 0.0025 | 1.6 | 6 | 0.4 | 2.4 | 1.1 | 4.0 | 1.1 | 0.5 | 2.7 | 1.3 |
| <i>s_i</i> : | 0.03 | 0.05 | 0.0024 | 0.5 | 1.4 | 0.21 | 1.1 | 0.6 | 1.5 | 0.4 | 0.4 | 0.7 | 0.3 |

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 5 but usually 6 single values of one laboratory. " < "-values have not been considered in statistical evaluation.

M : mean of means of data sets

s_M : standard deviation of means of data sets

s_i : mean of standard deviations of data sets under repeatability conditions

numbers in *italics* are indicative values

Mass fraction in mg/kg (indicative values)

| Line No. | Al | Cd | Cr | Co | Mg | Se | Si | Te | Ti | Zr |
|----------|------|-------|-------|-------|-------|------|------|-------|-------|-------|
| 1 | 0.9 | 0.19 | 1 | 0.23 | 0.9 | 1.0 | < 10 | 0.32 | 0.18 | 1 |
| 2 | < 1 | < 1 | < 0.5 | 0.38 | < 0.3 | 1.5 | < 10 | < 0.5 | < 0.5 | < 0.5 |
| 3 | < 1 | < 1 | < 0.5 | 0.38 | < 1 | 1.5 | | < 1 | < 0.7 | < 0.5 |
| 4 | < 10 | < 1 | < 1 | 0.9 | < 1 | < 1 | | | < 1 | < 2 |
| 5 | | < 1 | < 1 | < 0.5 | < 2 | < 20 | | | < 10 | |
| 6 | | < 1 | < 1 | < 1 | < 5 | | | | | |
| 7 | | < 1 | < 1 | < 1 | | | | | | |
| 8 | | < 1.5 | < 2 | < 1 | | | | | | |
| 9 | | | | < 1.5 | | | | | | |
| 10 | | | | < 5 | | | | | | |

ANALYTICAL METHOD USED FOR CERTIFICATION

| Element | Line no. | Method |
|---------|-----------------|--|
| Cu | 2, 4, 5, 8, | Electrogravimetry |
| | 3 | X-ray fluorescence analysis |
| | 1, 6, 7, 9 | Electrogravimetry, separation of Sn |
| Sn | 1 | PAA |
| | 2, 5, 6, 10, 11 | ICP OES |
| | 3, 7 | Iodometric titration |
| | 4, 8 | FAAS |
| | 9 | X-ray fluorescence analysis |
| Ag | 1, 2 | ICP OES |
| | 3, 7, 9 | FAAS |
| | 4 | ICP OES, separation of Sn |
| | 5 | PAA |
| | 6 | IDMS |
| | 8 | NAA |
| | 10 | ICP OES, separation of Sn, electrolytic separation of Cu |
| Fe | 1, 2, 3, 6, 8 | ICP OES |
| | 4 | FAAS, separation of Sn, electrolytic separation of Cu |
| | 5 | Photometric with 1,10-Phenanthroline |
| | 7, 12 | FAAS |
| | 9, 13, 14 | ICP OES, separation of Sn, electrolytic separation of Cu |
| | 10 | ICP OES, separation of Sn |
| | 11 | IDMS |

| | | |
|----|-----------------|--|
| Mn | 1, 2 | FAAS, separation of Sn, electrolytic separation of Cu |
| | 3 | ET AAS, separation of Sn, La(OH) ₃ -precipitation |
| | 4, 9 | FAAS |
| | 5, 7, 8, 10, 12 | ICP OES |
| | 6, 13, 14 | ICP OES, separation of Sn, electrolytic separation of Cu |
| | 11 | ICP OES, separation of Sn |
| Ni | 1, 12 | FAAS, separation of Sn, electrolytic separation of Cu |
| | 2, 5, 7, 8, 15 | ICP OES |
| | 3, 6, 13 | FAAS |
| | 4, 14 | ICP OES, separation of Sn, electrolytic separation of Cu |
| | 9 | ICP OES, separation of Sn |
| | 10 | IDMS |
| | 11 | Photometric with Diacetyldioxime, extraction |
| | 16 | PAA |
| P | 1, 2, 4 | Photometric as phosphovanadomolybdate, without extraction |
| | 3, 5, 7, 8 | ICP OES |
| | 6 | Photometric as phosphovanadomolybdate, after extraction |
| Pb | 1, 2, 10 | ICP OES |
| | 3 | ET AAS, La(OH) ₃ -precipitation |
| | 4 | IDMS |
| | 5, 9 | FAAS |
| | 6, 8 | FAAS, separation of Sn, electrolytic separation of Cu |
| | 7 | ET AAS, separation of Sn, La(OH) ₃ -precipitation |
| Zn | 1 | NAA |
| | 2, 3, 5 | ICP OES |
| | 4, 8, 10, 15 | FAAS |
| | 6 | ICP OES, electrolytic separation of Cu |
| | 7 | ICP OES, separation of Sn |
| | 9 | IDMS |
| | 11, 12 | ICP OES, separation of Sn, electrolytic separation of Cu |
| | 13, 14 | FAAS, separation of Sn, electrolytic separation of Cu |
| Al | 1, 3 | ICP OES |
| | 2 | ICP OES, separation of Sn |
| | 4, 5 | ICP OES, separation of Sn, electrolytic separation of Cu |
| As | 1 | Photometric as Molybdenum Blue after extraction |
| | 2 | ICP OES |
| | 3 | ET AAS |
| | 4 | FAAS, separation of Sn, electrolytic separation of Cu |
| | 5 | PAA |
| | 6 | NAA |
| | 7 | ICP OES, separation of Sn, electrolytic separation of Cu |
| Bi | 1, 2 | ET AAS |
| | 3 | FAAS, separation of Sn, electrolytic separation of Cu |
| Cd | 1, 3, 5, 8 | ICP OES |
| | 2, 4, 7 | ICP OES, separation of Sn, electrolytic separation of Cu |

| | | |
|----|---------------------------------------|---|
| Co | 1, 5, 8, 10 2 3, 4, 6, 9 7 | ICP OES, separation of Sn, electrolytic separation of Cu NAA ICP OES FAAS, separation of Sn, electrolytic separation of Cu |
| Cr | 1, 3 2 4 5, 8 6 7 9 | ICP OES ICP OES, separation of Sn PAA ICP OES, separation of Sn, electrolytic separation of Cu FAAS, separation of Sn, electrolytic separation of Cu FAAS NAA |
| Mg | 1, 2 3 4 5, 6 | ICP OES ICP OES, separation of Sn FAAS ICP OES, separation of Sn, electrolytic separation of Cu |
| S | 1, 4 2 3 5 | Infrared absorption after combustion Microtitration of sulphide ICP OES Photometric determination of H ₂ S as Molybdenum Blue |
| Sb | 1 2 3 4 5 6 | ET AAS ET AAS, La(OH) ₃ -precipitation ICP OES, separation of Sn, electrolytic separation of Cu NAA ICP OES Photometric with Rhodamine B |
| Se | 1, 3 2 4 5 | ICP OES NAA FAAS PAA |
| Si | 1, 2 | ICP OES |
| Te | 1 2 3 | ET AAS with Se as collector precipitate ET AAS FAAS, separation of Sn, electrolytic separation of Cu |
| Ti | 1, 3, 4 2 5 | ICP OES ICP OES, separation of Sn PAA |
| Zr | 1, 3 2 4 | ICP OES ICP OES, separation of Sn PAA |

Abbreviations:

ET AAS: Electrothermal Atomic Absorption Spectrometry
 FAAS: Flame Atomic Absorption Spectrometry
 ICP-MS: Inductively Coupled Plasma - Mass Spectrometry
 ICP OES: Inductively Coupled Plasma - Optical Emission Spectrometry
 IDMS: Isotope Dilution Mass Spectrometry
 NAA: Neutron Activation Analysis
 PAA: Photon Activation Analysis

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INSTRUCTIONS FOR USE

Before use, the surface of the material must be cleaned by turning on a lathe.

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[®] - EB374 is available on request.

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