

CERTIFICATE OF ANALYSIS

ERM[®]-EB377**CuSn6**

| Certified Values | | |
|------------------|-------------------------------|---------------------------|
| Element | Certified value ¹⁾ | Uncertainty ²⁾ |
| | Mass fraction in % | |
| Cu | 94.04 | ± 0.05 |
| Sn | 5.92 | ± 0.13 |
| | Mass fraction in mg/kg | |
| Ag | 64.4 | ± 1.1 |
| Al | 45.1 | ± 1.2 |
| Bi | 42.2 | ± 1.5 |
| Cr | 66.9 | ± 2.1 |
| Fe | 104.2 | ± 2.7 |
| Mn | 92.1 | ± 2.1 |
| Ni | 107.4 | ± 1.5 |
| Pb | 44.9 | ± 2.3 |
| Sb | 13.0 | ± 1.3 |
| Se | 55 | ± 4 |
| Zn | 100.6 | ± 3.0 |

¹⁾ Unweighted mean value of the means of accepted sets of data (at least 5 usually 6), 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 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[®]-EB377 was originally certified as BAM-377. 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>).

Accepted as an ERM[®], Berlin, 2004-04-14.

Berlin,

BAM Berlin
Department I
Analytical Chemistry;
Reference Materials
12200 Berlin, Germany

BAM Berlin
Division I.1
Inorganic Chemical Analysis;
Reference Materials
12200 Berlin, Germany

Prof. Dr. I. Nehls
(Head of Department)

Dr. R. Matschat
(Head of Division)

| Indicative Values³⁾ | | |
|---------------------------------------|--------------------------------|---------------------------|
| | Indicative value ⁴⁾ | Uncertainty ⁵⁾ |
| Element | Mass fraction in mg/kg | |
| S | 6.8 | ± 0.8 |
| Element | Mass fraction in mg/kg | |
| Cd, Mg, Te, Ti | < 1 | |
| Co | < 2 | |
| As, P | < 10 | |
| Si | 134 | |

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.

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.

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.

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

Mass fraction in %

Mass fraction in mg/kg

| Line No.. | Cu | Sn | Ag | Al | Bi | Cr | Fe | Mn | Ni | Pb | Sb | Se | Zn |
|------------------------|-------|------|------|------|------|------|-----|------|-------|------|------|------|-------|
| 1 | 93.94 | 5.84 | 61.7 | 43.4 | - | - | - | - | - | 39.8 | 10.8 | - | 90.1 |
| 2 | 93.98 | 5.85 | 63.3 | 44.4 | 41.0 | 60.0 | 98 | 87.4 | 104.3 | 41.0 | 11.7 | 48.5 | 93.0 |
| 3 | 94.01 | 5.87 | 63.7 | 44.9 | 41.4 | 62.2 | 98 | 88.3 | 104.4 | 44.5 | 12.0 | 50.2 | 95.1 |
| 4 | 94.06 | 5.89 | 64.3 | 45.0 | 41.5 | 62.3 | 100 | 89.4 | 105.8 | 44.9 | 12.9 | 52.4 | 96.7 |
| 5 | 94.07 | 5.90 | 64.6 | 46.0 | 41.5 | 64.9 | 102 | 90.1 | 106.5 | 45.4 | 13.0 | 55.8 | 97.9 |
| 6 | 94.09 | 5.91 | 64.9 | 46.3 | 43.5 | 65.0 | 102 | 90.2 | 106.6 | 46.4 | 14.1 | 55.9 | 98.3 |
| 7 | 94.09 | 5.92 | 65.4 | - | 44.4 | 66.0 | 105 | 90.5 | 106.8 | 46.7 | 14.7 | 58.2 | 100.4 |
| 8 | 94.10 | 5.93 | 65.7 | | | 66.6 | 106 | 91.1 | 106.9 | 47.8 | 14.9 | 58.5 | 100.6 |
| 9 | 94.14 | 5.98 | 65.8 | | | 67.4 | 107 | 91.2 | 106.9 | 48.1 | | 58.8 | 100.6 |
| 10 | | 5.99 | - | | | 68.0 | 107 | 92.8 | 107.3 | - | | | 103.7 |
| 11 | | 6.01 | | | | 68.3 | 108 | 95.4 | 108.0 | | | | 104.3 |
| 12 | | | | | | 68.6 | 108 | 96.2 | 108.2 | | | | 105.8 |
| 13 | | | | | | 68.6 | 111 | 97.2 | 110.8 | | | | 106.3 |
| 14 | | | | | | 70.3 | | 97.3 | 113.6 | | | | 106.7 |
| 15 | | | | | | 72.2 | | | - | | | | 109.0 |
| 16 | | | | | | 73.1 | | | | | | | |
| <i>M</i> : | 94.04 | 5.92 | 64.4 | 45.0 | 42.2 | 66.9 | 104 | 92.1 | 107.4 | 44.9 | 13.0 | 55 | 101 |
| <i>s_M</i> : | 0.06 | 0.06 | 1.3 | 1.1 | 1.4 | 3.7 | 4 | 3.4 | 2.5 | 2.9 | 1.5 | 4 | 6 |
| <i>s_i</i> : | 0.04 | 0.05 | 0.9 | 1.6 | 1.2 | 1.2 | 2.2 | 1.6 | 1.7 | 1.0 | 0.7 | 1.6 | 2.1 |

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. | As | Cd | Co | Mg | P | S | Si | Te | Ti |
|----------|------|-------|-------|-------|------|-----|-----|-------|-----|
| 1 | 1 | < 1 | 0.14 | 0.7 | 5 | 6.5 | 121 | 0.6 | 0.3 |
| 2 | < 1 | < 1 | 0.3 | 0.7 | < 2 | 6.5 | 147 | < 0.5 | < 1 |
| 3 | < 3 | < 1 | 2.0 | < 0.3 | < 10 | 6.7 | | < 1 | < 1 |
| 4 | < 3 | < 1 | < 1 | < 1 | < 10 | 7.5 | | | |
| 5 | < 10 | < 1 | < 1 | < 2 | < 10 | < 5 | | | |
| 6 | < 10 | < 1 | < 1 | < 5 | | | | | |
| 7 | | < 1.5 | < 1.5 | | | | | | |
| 8 | | | < 5 | | | | | | |
| 9 | | | | | | | | | |
| 10 | | | | | | | | | |

ANALYTICAL METHOD USED FOR CERTIFICATION

| Element | Line no. | Method |
|---------|----------------|--|
| Cu | 1 | X-ray fluorescence analysis |
| | 2, 7, 9 | Electrogravimetry, separation of Sn |
| | 3, 4, 5, 6, 8 | Electrogravimetry |
| Sn | 1, 2 | Iodometric titration |
| | 3, 5, 6, 9, 10 | ICP OES |
| | 4, 8 | FAAS |
| | 7 | X-ray fluorescence analysis |
| | 11 | PAA |
| Ag | 1, 2 | ICP OES |
| | 3 | PAA |
| | 4 | NAA |
| | 5 | IDMS |
| | 6, 8, 9 | FAAS |
| | 7 | FAAS, separation of Sn |
| | 10 | ICP OES, separation of Sn, electrolytic separation of Cu |
| Al | 1, 2, 4, 5 | ICP OES |
| | 3, 6 | ICP OES, separation of Sn, electrolytic separation of Cu |
| | 7 | Photometric with Chromazurol S |
| Bi | 1 | FAAS, separation of Sn, La(OH) ₃ -precipitation |
| | 2 | FAAS, electrolytic separation of Cu |
| | 3 | ICP OES |
| | 4, 7 | FAAS |
| | 5, 6 | ET AAS |

| | | |
|----------------|------------------|--|
| Cr | 1, 4 | FAAS, separation of Sn, electrolytic separation of Cu |
| | 2, 5 | FAAS |
| | 3, 10, 13, 16 | ICP OES, separation of Sn, electrolytic separation of Cu |
| | 6 | PAA |
| | 7, 9, 11, 14, 15 | ICP OES |
| | 8 | NAA |
| | 12 | IDMS |
| | 1 | ICP OES |
| Fe | 1, 3, 4, 8, 10 | ICP OES, separation of Sn, electrolytic separation of Cu |
| | 2, 12 | FAAS, separation of Sn, La(OH) ₃ -precipitation |
| | 5 | FAAS, electrolytic separation of Cu |
| | 6 | FAAS, separation of Sn, electrolytic separation of Cu |
| | 7 | FAAS |
| | 9, 11 | FAAS |
| | 13 | IDMS |
| Mn | 1, 13 | FAAS, separation of Sn, electrolytic separation of Cu |
| | 2, 11, 14 | ICP OES, separation of Sn, electrolytic separation of Cu |
| | 3 | FAAS, electrolytic separation of Cu |
| | 4, 6, 8, 10, 12 | ICP OES |
| | 5, 7 | FAAS |
| | 9 | PAA |
| | Ni | 1, 3, 13 |
| 2, 4, 8, 9, 10 | | ICP OES |
| 5 | | FAAS, electrolytic separation of Cu |
| 6, 15 | | FAAS, separation of Sn, electrolytic separation of Cu |
| 7, 12 | | FAAS |
| 11 | | IDMS |
| 14 | | PAA |
| Pb | 1 | FAAS, separation of Sn, La(OH) ₃ -precipitation |
| | 2 | FAAS, electrolytic separation of Cu |
| | 3, 5, 9 | FAAS |
| | 4, 7, 8 | ICP OES |
| | 6 | IDMS |
| | 10 | FAAS, separation of Sn, electrolytic separation of Cu |
| | 11 | ICP OES, separation of Sn, electrolytic separation of Cu |
| Sb | 1 | ET AAS |
| | 2 | FAAS, electrolytic separation of Cu |
| | 3 | ET AAS, La(OH) ₃ -precipitation |
| | 4 | PAA |
| | 5 | NAA |
| | 6 | Photometric with Rhodamine B |
| | 7 | FAAS |
| | 8 | ICP OES |
| Se | 1 | Photometrie |
| | 2 | PAA |
| | 3 | FAAS, electrolytic separation of Cu |
| | 4 | NAA |
| | 5, 6, 8, 9 | ICP OES |
| | 7 | ET AAS |

| | | | |
|----|-------------|--|--|
| Zn | 1 | NAA | |
| | 2, 4, 5 | ICP OES | |
| | 3, 8, 9, 15 | FAAS, | |
| | 6 | ICP OES, electrolytic separation of Cu | |
| | 7 | IDMS | |
| | 10 | FAAS, separation of Sn, electrolytic separation of Cu | |
| | 11, 12, 13 | ICP OES, separation of Sn, electrolytic separation of Cu | |
| | 14 | FAAS, electrolytic separation of Cu | |
| | As | 1 | ET AAS, with Se as collector precipitate |
| | | 2 | FAAS, electrolytic separation of Cu |
| 3 | | PAA | |
| 4 | | NAA | |
| 5 | | ICP OES, separation of Sn, electrolytic separation of Cu | |
| 6 | | ICP OES | |
| Cd | 1, 2, 3, 7 | ICP OES | |
| | 4, 5 | ICP OES, separation of Sn, electrolytic separation of Cu | |
| | 6 | FAAS, electrolytic separation of Cu | |
| Co | 1 | NAA | |
| | 2, 3, 4, 7 | ICP OES | |
| | 5 | FAAS, electrolytic separation of Cu | |
| | 6, 8 | ICP OES, separation of Sn, electrolytic separation of Cu | |
| Mg | 1, 2, 3 | ICP OES | |
| | 4 | FAAS, electrolytic separation of Cu | |
| | 5, 6 | ICP OES, separation of Sn, electrolytic separation of Cu | |
| P | 1, 3, 4 | ICP OES | |
| | 2, 5 | Photometric as phosphovanadomolybdate | |
| S | 1 | Photometric determination of H ₂ S as Molybdenum Blue | |
| | 2 | ICP OES | |
| | 3 | Microtitration of sulphide | |
| | 4 | Coulometry after combustion | |
| | 5 | Infrared absorption after combustion | |
| Si | 1, 2 | ICP OES | |
| Te | 1 | ET AAS, with Se as collector precipitate | |
| | 2 | ET AAS | |
| | 3 | FAAS, electrolytic separation of Cu | |
| Ti | 1, 2, 3 | ICP OES | |

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

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

PARTICIPANTS

Bundesanstalt für Materialforschung und -prüfung, Berlin (Germany)
Hüttenwerke Kayser AG, Lünen (Germany)
KM Europa Metal AG, Osnabrück (Germany)
Krupp VDM GmbH, Werdohl (Germany)
Labor Peter Rohmann, Lutherstadt Eisleben (Germany)
Mansfelder Kupfer und Messing GmbH-Nord, Hettstedt (Germany)
Mansfelder Kupfer und Messing GmbH-Süd, Hettstedt (Germany)
Max-Planck-Institut für Metallforschung, Stuttgart (Germany)
Norddeutsche Affinerie AG, Hamburg (Germany)
Sundwiger Messingwerk, Hemer (Germany)
Wieland-Werke AG, Ulm (Germany)

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[®]-EB377 is available on request.

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

Richard-Willstätter-Straße 11, 12489 Berlin, Germany

Phone: +49 30 8104 2061

e-mail: sales.crm@bam.de

Fax: +49 30 8104 1117

internet: www.bam.de