

Certificate for Primary Reference Material**BAM-Y005****high purity tin****(BAM-A-primary-Sn-1)****Certified quantity value**

When applying the prescribed sample treatment as specified below, the mass fraction w of tin in material BAM-Y005 is:

| Element | Mass fraction w in kg/kg | Uncertainty in kg/kg |
|----------------|--|----------------------------------|
| Tin | 0,999 91 | $\pm 0,000 06$ |

The uncertainty given is an expanded uncertainty $U=k \cdot u_c$ with $k=2$ evaluated according the GUM [1].

Taking the IUPAC value [2] for the atomic weight of tin to be $(118,710 \pm 0,007)$, the value certified corresponds to an amount-of-substance of tin content of $k(\text{Sn}) = w/M = (8,423 1 \pm 0,001 1) \text{ mol/kg}$ (expanded uncertainty $U=k \cdot u_c$ with $k=2$) in the solid material.

The certified value has been established by subtracting the sum of the mass fractions of 91 possible impurities with other elements from the value for ideal purity of 1,000 000 kg/kg.

73 of 91 values of possible impurities with other elements are based on measurements. The sum of the mass fractions of the measured impurities is $w_{\Sigma}(\text{measured values}) = (42 \pm 9) \text{ mg/kg}$.

From theoretical considerations very reasonable estimates have been made for the upper limits of the impurities with noble gases and artificial/instable elements. The sum of the mass fractions of these 12 of 91 possible impurities is $w_{\Sigma}(\text{theoretical values}) = (0,26 \pm 0,25) \text{ mg/kg}$. The influence of this value on the certified value and its associated uncertainty is marginal.

The impurities of H, Si, F, Cl, Br and I (6 of 91 possible impurities) have not been measured yet. For these impurities upper limit values have been conservatively estimated. The sum of the mass fractions of the impurities estimated is $w_{\Sigma}(\text{estimated values}) = (50 \pm 27) \text{ mg/kg}$. The influence of this value on the certified value and its associated uncertainty is significant.

Note, that the values for $w_{\Sigma}(\text{measured values})$, $w_{\Sigma}(\text{theoretical$

Note: In line with the ISO 31-0 (1992) in this document the comma (and not the dot) is used as a decimal separator.

values) and w_2 (estimated values) are not certified but only of informative nature. The uncertainties given for these three values are combined uncertainties u_c .

Safety instructions: Avoid skin contact and incorporation. Handle with caution - as any chemical substance.

Sample description: The material is provided in glass bottles containing about 0,5 g of compact material.

Recommendation for correct storing: The bottle should be kept closed at ambient conditions and in a clean and dry environment.

Expiration of certificate: This certificate expires formally ten years after seal affixation on the certificate. (The material itself is assumed to be stable > 200 years).

Prescribed sample treatment

Before use, the material has to be etched in order to achieve the defined state, to which the certified quantity value applies. Solvents of high purity (at least p.a. grade) must be used. The material must be processed (i.e. weighed) within 1 h after the final etching.

T1. The preparation has to be performed under clean air conditions and using high purity acids, high purity water and purified vessels.

T2. Sub-sample masses of $m > 0,2$ g and a surface to mass ratio $A/m < 14$ cm²/g must be used.

T3. Etch individual metal pieces for 1 min in 20 mL (98 % acetic acid + 65 % HNO₃ + 40 % HF) (75 Vol. + 25 Vol. + 1,5 Vol.) while moving the solution at room temperature. Use the solution only once. Rinse in 3 × 30 mL H₂O (in an ultrasonic bath; use water only once).

T4. Etch individual metal pieces for 1 min in 20 mL (85 % H₃PO₄ + 40 % HF) (25 Vol. + 2 Vol.) while moving the solution at room temperature. Use the solution only once. Rinse in 3 × 30 mL H₂O (in an ultrasonic bath; use water only once).

T5. Rinse 3× in CH₃OH; expose 1 min to hair drier or 10 min in Ar-flow.

T6. The sample can then mechanically be cut using clean cutting tools. Avoid surface contamination and reoxidation.

T7. Process material again as described in T3 but with an etching time of 4 min. Process material as in T4 and -T5. The material must then have a shiny metallic surface, free of white spots.

Obey the safety instructions of the chemicals and equipment used!

Notes

N01. The certified quantity value with associated measurement uncertainty carried by BAM-Y005, is traceable to the related units of the Système International d'Unités (SI).

N02. The certified value with associated measurement uncertainty carried by BAM-Y005, is a realisation of the unit for mass fraction measurements of tin. Using the concept of molar mass and gravimetry, it is a realisation of the unit for amount-of-substance content (amount-of-substance per mass) measurements of tin.

N03. In co-operation with Physikalisch Technische Bundesanstalt (PTB), BAM-Y005 is the primary National Measurement Standard of Germany for the measurement of tin.

N04. BAM-Y005 is intended to be used to prepare gravimetrically secondary measurement standards (solid or solution).

N05. The material is intended for metrological use within National and International Metrology Institutes only.

N06. Technical details about the certification can be found in the 'Certification Report BAM-Y005', which is available from BAM on request and at www.bam.de.

N07. The certified value with associated measurement uncertainty has been established by measuring the impurities in a high purity tin material using a variety of measurement methods. The quantity values for the individual impurities are given as additional information in Table 1. These quantity values are not certified values.

N08. All measurements, which were used for the certification of BAM-Y005 have been performed by BAM laboratories.

Abbreviations

| | |
|-------------|---|
| HE | Carrier gas hot extraction and combustion analysis |
| HR-ICP-MS | High resolution mass spectrometry using inductively coupled plasma |
| ICP-MS | Mass spectrometry using inductively coupled plasma |
| ML-ICP-MS | Mass spectrometry using inductively coupled plasma in medium resolution |
| THE | Estimated value based on theoretical considerations |
| U | Expanded measurement uncertainty |
| u_c | Combined measurement uncertainty |
| ULV | Upper limit value based on measurements |
| ULV-EST | Upper limit value based on estimate; not yet measured |
| unc. contr. | Parameter characterising the relative influence of the uncertainty of an individual impurity mass fraction to the combined uncertainty of the tin mass fraction calculated according to: $u^2(w_i) / \sum u^2(w_j)$ |

Table 1: The individual values used to establish the certified value. Note, that the values given here are not certified, but only of informative nature!

| method | w | u(w) | unc. | method | w | u(w) | unc. | method | w | u(w) | unc. |
|------------|---------|--------|---------|--------------|---------|--------|---------|--|---------|--------|---------|
| | mg/kg | mg/kg | contr. | | mg/kg | mg/kg | contr. | | mg/kg | mg/kg | contr. |
| H EST-ULV | < 50 | 25 | 0,76 | Ge ICP-MS | < 0,1 | 0,05 | << 0,01 | Eu ICP-MS | < 0,005 | 0,0025 | << 0,01 |
| He THE | < 0,001 | 0,0005 | << 0,01 | As ICP-MS | < 0,2 | 0,1 | << 0,01 | Gd ICP-MS | < 0,05 | 0,025 | << 0,01 |
| Li ICP-MS | < 3 | 1,5 | << 0,01 | Se ICP-MS | < 2,5 | 1,25 | << 0,01 | Tb ICP-MS | < 0,005 | 0,0025 | << 0,01 |
| Be ICP-MS | < 0,5 | 0,25 | << 0,01 | Br EST-ULV | < 10 | 5 | 0,03 | Dy ICP-MS | < 0,001 | 0,0005 | << 0,01 |
| B ICP-MS | < 1 | 0,5 | << 0,01 | Kr THE | < 0,001 | 0,0005 | << 0,01 | Ho ICP-MS | < 0,01 | 0,005 | << 0,01 |
| C HE | < 5 | 2,5 | 0,01 | Rb ICP-MS | < 0,01 | 0,005 | << 0,01 | Er ICP-MS | < 0,001 | 0,0005 | << 0,01 |
| N HE | < 3,9 | 1,95 | << 0,01 | Sr ICP-MS | < 0,01 | 0,005 | << 0,01 | Tm ICP-MS | < 0,005 | 0,0025 | << 0,01 |
| O HE | < 8,4 | 4,2 | 0,02 | Y ICP-MS | < 0,005 | 0,0025 | << 0,01 | Yb ICP-MS | < 0,01 | 0,005 | << 0,01 |
| F EST-ULV | < 10 | 5 | 0,03 | Zr ICP-MS | < 0,01 | 0,005 | << 0,01 | Lu ICP-MS | < 0,005 | 0,0025 | << 0,01 |
| Ne THE | < 0,001 | 0,0005 | << 0,01 | Nb ICP-MS | < 0,005 | 0,0025 | << 0,01 | Hf ICP-MS | < 0,005 | 0,0025 | << 0,01 |
| Na ICP-MS | < 2 | 1 | << 0,01 | Mo ICP-MS | < 0,05 | 0,025 | << 0,01 | Ta ICP-MS | < 0,01 | 0,005 | << 0,01 |
| Mg ICP-MS | < 0,1 | 0,05 | << 0,01 | Tc THE | < 0,001 | 0,0005 | << 0,01 | W ICP-MS | < 1 | 0,5 | << 0,01 |
| Al ICP-MS | < 0,1 | 0,05 | << 0,01 | Ru ICP-MS | < 0,01 | 0,005 | << 0,01 | Re ICP-MS | < 0,005 | 0,0025 | << 0,01 |
| Si EST-ULV | < 10 | 5 | 0,03 | Rh ICP-MS | < 0,005 | 0,0025 | << 0,01 | Os ICP-MS | < 0,01 | 0,005 | << 0,01 |
| P ICP-MS | < 5 | 2,5 | 0,01 | Pd ICP-MS | < 0,01 | 0,005 | << 0,01 | Ir ICP-MS | < 0,005 | 0,0025 | << 0,01 |
| S HE | < 3,3 | 1,65 | << 0,01 | Ag ICP-MS | < 0,01 | 0,005 | << 0,01 | Pt ICP-MS | < 0,01 | 0,005 | << 0,01 |
| Cl EST-ULV | < 10 | 5 | 0,03 | Cd ML-ICP-MS | < 0,5 | 0,25 | << 0,01 | Au ICP-MS | < 0,005 | 0,0025 | << 0,01 |
| Ar THE | < 0,50 | 0,25 | << 0,01 | In ML-ICP-MS | < 5 | 2,5 | 0,01 | Hg ICP-MS | < 0,1 | 0,05 | << 0,01 |
| K ICP-MS | < 2 | 1 | << 0,01 | Sn matrix | | | | Tl ICP-MS | < 0,05 | 0,025 | << 0,01 |
| Ca ICP-MS | < 2,5 | 1,25 | << 0,01 | Sb ML-ICP-MS | < 5 | 2,5 | 0,01 | Pb ICP-MS | 7,1 | 2,13 | 0,01 |
| Sc ICP-MS | < 0,1 | 0,05 | << 0,01 | Te ML-ICP-MS | < 1 | 0,5 | << 0,01 | Bi ICP-MS | 0,06 | 0,018 | << 0,01 |
| Ti ICP-MS | < 0,1 | 0,05 | << 0,01 | I EST-ULV | < 10 | 5 | 0,03 | Po THE | < 0,001 | 0,0005 | << 0,01 |
| V ICP-MS | < 0,05 | 0,025 | << 0,01 | Xe THE | < 0,001 | 0,0005 | << 0,01 | At THE | < 0,001 | 0,0005 | << 0,01 |
| Cr ICP-MS | < 0,1 | 0,05 | << 0,01 | Cs ML-ICP-MS | < 5 | 2,5 | 0,01 | Rn THE | < 0,001 | 0,0005 | << 0,01 |
| Mn ICP-MS | < 0,1 | 0,05 | << 0,01 | Ba ML-ICP-MS | < 5 | 2,5 | 0,01 | Fr THE | < 0,001 | 0,0005 | << 0,01 |
| Fe ICP-MS | 0,9 | 0,27 | << 0,01 | La ML-ICP-MS | < 2 | 1 | << 0,01 | Ra THE | < 0,001 | 0,0005 | << 0,01 |
| Co ICP-MS | < 0,05 | 0,025 | << 0,01 | Ce ICP-MS | < 0,05 | 0,025 | << 0,01 | Ac ICP-MS | < 0,01 | 0,005 | << 0,01 |
| Ni ICP-MS | < 0,5 | 0,25 | << 0,01 | Pr ML-ICP-MS | < 1 | 0,5 | << 0,01 | Th ICP-MS | < 0,001 | 0,0005 | << 0,01 |
| Cu ICP-MS | < 0,05 | 0,025 | << 0,01 | Nd ICP-MS | < 0,005 | 0,0025 | << 0,01 | Pa ICP-MS | < 0,001 | 0,0005 | << 0,01 |
| Zn ICP-MS | < 0,1 | 0,05 | << 0,01 | Pm THE | < 0,001 | 0,0005 | << 0,01 | U ICP-MS | < 0,01 | 0,005 | << 0,01 |
| Ga ICP-MS | < 0,25 | 0,125 | << 0,01 | Sm ICP-MS | < 0,01 | 0,005 | << 0,01 | Value contributes > 10 % to combined uncertainty | | | |

References

1 Guide to the Expression of Uncertainty in measurement, ISO, Geneva, (1993).

2 IUPAC: 'Atomic weights of the elements 1995' Pure & Appl. Chem. 68 (1996), 2339-2359.

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