

Certificate

Produced in double accredited
laboratory fulfilling
ISO/IEC 17025 and
ISO Guide 34

This certificate is designed in accordance with ISO Guide 31^[1].

Object of certification: **Antimony standard for AAS**

Fluka Product No.: 94117 (Lot 1409575, Filling code 53808196)

Composition: Antimony semimetal (pure material) in 2% HNO₃ and <0.1% HF (prepared from HNO₃ TraceSELECT[®], HF TraceSELECT[®] and water TraceSELECT[®]Ultra, 18.2 MΩ·cm, 0.22 μm filtered)

Density at 20°C: $\rho = 1010 \text{ kg m}^{-3}$ $u_c(\rho) = 0.5 \text{ kg m}^{-3}$

Intended use: Calibration of AAS, ICP-spectrometry, spectrophotometry or any other analytical technique

Storing and handling: This reference material shall be stored between 5°C and 30°C. The bottle's temperature must be 20°C and shaken well before every use. If storage of a partially used bottle is necessary, the cap should be tightly sealed and the bottle should be stored at reduced temperature (e.g. refrigerator) to minimize transpiration rate.

Expiry date: **3. September 2011**

Bottle opening date: 

Certified value traceable to SI unit kg and uncertainty according to ISO Guide 35 ^[2] and Eurachem/CITAC Guide ^[3]		
Constituent	Certified value at 20°C ^[4]	Expanded uncertainty [$U = k u_c$; $k = 2$]
Antimony	999 mg L⁻¹	4 mg L⁻¹
	989 mg kg⁻¹	4 mg kg⁻¹

1. CONCEPT OF CERTIFICATION AND TRACEABILITY STATEMENT

To guarantee top reliability of the values for this TraceCERT[®] certified reference material three independent procedures were followed. The values have to agree in the range of their uncertainties, but the impurity corrected value from the gravimetric preparation has been chosen as certified value^[5].

1. Gravimetric preparation using pure materials is a practical realization of concentration units, through conversion of mass to amount of substance^[5]. If the purity of the materials is demonstrated and if contamination and loss of material is strictly prevented this approach allows highest accuracy and small uncertainties. The certified value of TraceCERT[®] reference materials is based on this approach and directly traceable to the SI unit kilogram. Therefore comprehensively characterized materials of high purity are used. All balances are certified by DKD and calibrated with OIML Class E2 (up to 12 kg) and F2 (up to 64 kg) weights.
2. The starting material is measured against a certified reference material (i.e. NIST, BAM or EMPA) followed by gravimetric preparation using balances calibrated with SI-traceable weights. Consequently the value calculated by this unbroken chain of comparisons is traceable to the reference to which the starting material is compared.
3. Whenever applicable the bottled TraceCERT[®] calibration solution is compared to a second reference (e.g. from NIST, BAM or EMPA) which is independent from the first reference.

2. PURITY OF STARTING MATERIALS

For high purity materials ($P > 99.9\%$) the most appropriate way of purity determination is to quantify the impurities (w_i) and to subtract the sum from 100%. Impurities below the detection limit are considered with a contribution of half of the detection limit (DL_j).

$$P = 100\% - \sum_i w_i - \sum_j \left(\frac{DL_j}{2} \right)$$

Water containing materials were dried to absolute dryness by individual drying conditions (up to 600°C). When drying is impossible due to decomposition water was determined by high-precision KF-titration under supervision of H. Hoffmann at Sigma-Aldrich Laborchemikalien, Seelze (Germany). Water *TraceSELECT*® Ultra (18.2 MΩ·cm; 0.22 μm filtered, all metallic traces at ng kg⁻¹-level) and acid in *TraceSELECT*® quality was used for preparation.

3. TRACEABILITY MEASUREMENTS

Only internationally accepted reference materials e.g. from NIST (USA), BAM (Germany) or EMPA – Material Science & Technology (Switzerland) have been carefully selected to provide the basis for traceability to the SI unit Mole. When no such reference is available, an elemental metal or an adequate salt of highest available purity is used to confirm traceability to this pure material (and therefore to the SI unit kg).

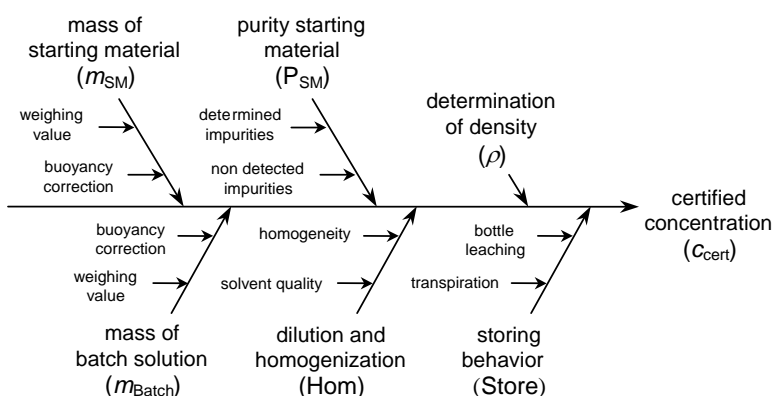
To underpin the certified gravimetric value all traceability measurements are performed with the most accurate and precise analytical technique available. Therefore titrimetry measurement series are applied whenever possible (corrected for trace impurities). When no titrimetric technique is available, the traceability measurements are performed with another analytical technique, e.g. ICP-OES or AAS.

Reference and applied technique used for traceability measurements of the starting material:
NIST SRM 3102a / ICP-OES






Reference and applied technique used for traceability measurements of the bottled solution:
NIST SRM 3102a / ICP-OES

4. UNCERTAINTY EVALUATION

All uncertainties are calculated according to Eurachem/CITAC Guide [3] and reported as combined expanded uncertainties at the 95% confidence level. For gravimetric preparation the uncertainty contributions are illustrated by the following cause-effect diagram [6]:



- Typical contributions:
- $u(m_{SM})$ < 0.01%
 - $u(m_{Batch})$ < 0.01%
 - $u(P_{SM})$ < 0.10%
 - $u(Hom)$ < 0.03%
 - $u(Store)$ < 0.17%
 - $u(\rho)$ < 0.05%
- Combined uncertainty [7]:
- $U_c(C_{cert})$ < 0.2%
- Expanded uncertainty:
- $U(C_{cert})$ < 0.4%

Certification laboratory	Certification body	Date of release	Quality systems
 J. Wuethrich	 K.-D. Schmidt, Ph.D.	October 10 th 2008	  

[1] ISO Guide 31, 1-7, 2nd Ed. (2000), "Reference materials - Contents of certificates and labels"
 [2] ISO Guide 35, 1-64, 3rd Ed. (2006), "Reference materials - General and statistical principles for certification"
 [3] Eurachem/CITAC Guide, 1-120, 2nd Ed. (2000), "Quantifying uncertainty in analytical measurement"
 [4] The mg kg⁻¹ value is the certified value whereas the mg L⁻¹ value is calculated with the density
 [5] Eurachem/CITAC Guide, 1-37, 1st Ed. (2003) "Traceability in chemical measurement"
 [6] Reichmuth, A., Wunderli, S., Weber, M., Meyer, V. R. (2004), The uncertainty of weighing data obtained with electronic analytical balances, *Microchimica Acta* 148: 133-141.
 [7] Calculated by combination of the squared contribution values