

# Certification Report – Certified Reference Material

## Certipur® AAS single element Standards

**Manufacturer:**

Merck KGaA, Frankfurter Str. 250, 64293 Darmstadt, Germany, Tel. +49(0)6151 720

**Accreditation:**

Merck KGaA, Darmstadt, Germany is accredited by the German accreditation authority as registered reference material producer (D-RM-15185-01-00) in accordance with **ISO 17034**.

**Introduction:**

This certification report contains additional information about certified values and uncertainties, homogeneity, stability, traceability and other relevant details of Certipur® AAS single element standards.

The scope of the ISO 17034 accreditation is documented in the annex to the accreditation certificate D-RM-15185-01-00. The AAS single element standards are produced, characterized and certified by the legal entity Merck KGaA, Darmstadt, Germany.

**Preparation and packaging of CRMs:**

Certipur® AAS single element standards are prepared gravimetrically from high purity raw materials in either high purity acids or high purity bases Suprapur® and diluted with filtered (0.22µm) high purity water (18MΩ). All balances are regularly calibrated with analytical weight sets traceable to primary weights by PTB (Physikalisch Technische Bundesanstalt). The density of each batch is measured in g/cm<sup>3</sup> at 20°C and is used to calculate the concentration in mg/l.

Certipur® AAS single element standards are delivered in 100 ml and 500 ml high-density-PE bottles. The label on the products is in accordance to ISO Guide 31 and covers product number, article description, lot number, expiry date and safety data information. Packaging material is subject to comprehensive stability studies according to ISO Guide 35.

**Homogeneity:**

Homogeneity studies were conducted in accordance to ISO Guide 35.

A representative number of samples packaged in their final form was chosen systematically (stratified over the whole batch) for assessment of the between bottle homogeneity. Measurements have been carried out according to DIN EN ISO/IEC 17025. Results from multiple samples of the chosen bottles were evaluated using a one-way ANOVA (Analysis of variance) approach.

Typical homogeneity contributions to uncertainty  $u_{\text{hom}}$  are in the range of 0.13% for all standards (100 ml and 500 ml bottles).

**Stability:**

Stability studies were conducted in accordance to ISO Guide 35.

Stability comprises long- term stability, which is associated with the storage behavior of the CRMs under recommended storage conditions as well as short-term stability that takes any extra effects due to transport of the products into account (quantification of transport effects that result from temperature variations during shipment).



### Long-term stability:

Long-term stability of the products was evaluated using the classical experimental layout. Samples were stored at the recommended storage conditions before measurement. Measurements were conducted gravimetrically (as transpiration of the matrix during storage may raise the elemental concentration over the course of time) and verified by ICP-OES analysis according to DIN EN ISO/IEC 17025. Results were evaluated using a linear regression model and slopes significantly different from zero are communicated to the user as correction factor for the certified value depending on the storage time. The uncertainty of the slope is included into the uncertainty budget as long-term stability contribution  $u_{lts}$ .

Typical long-term stability contributions to uncertainty  $u_{lts}$  are in the range of 0.009% for standards in 100 ml bottles and in the range of 0.004% for standards in 500 ml bottles.

### Short-term (transportation) stability:

Short-term stability was evaluated using the classical experimental layout. Samples were stored at elevated temperatures that may arise during shipment of the products before measurement. Measurements were conducted gravimetrically (as transpiration of the matrix during storage may raise the elemental concentration over the course of time) and verified by ICP-OES analysis according to DIN EN ISO/IEC 17025. Results were evaluated using a linear regression model and the slope as well as the uncertainty of the slope is included into the uncertainty budget as short-term stability contribution  $u_{sts}$ .

Typical short-term stability contributions to uncertainty  $u_{sts}$  are in the range of 0.003% for standards in 100 ml bottles and in the range of 0.001% for standards in 500 ml bottles.

### Stability monitoring:

Merck KGaA, Darmstadt, Germany will monitor all Certipur® AAS single element standards over the period of their certification (until expiry date). If substantive technical changes occur that affect the validity of certification, the customer will be notified.

### Characterisation:

Characterisation of Certipur® AAS single element standards is carried out by the accredited quality control (QC) laboratory at Merck KGaA, Darmstadt, Germany according to DIN EN ISO / IEC 17025 by inductively coupled plasma optical emission spectroscopy (ICP - OES).

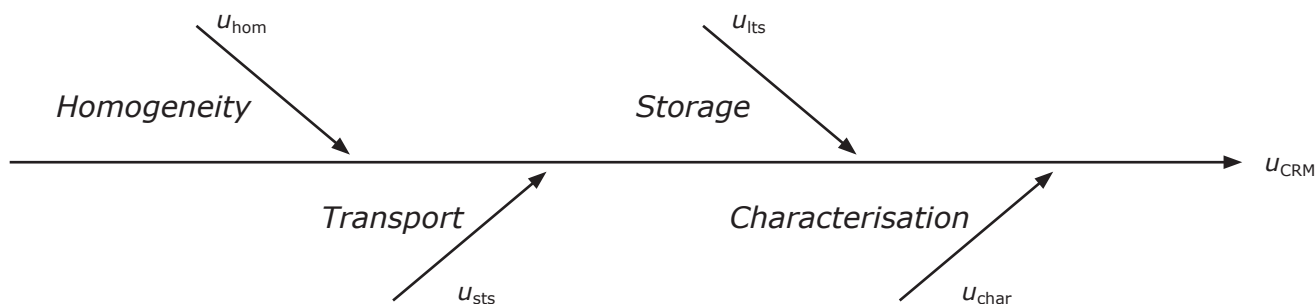
Typical characterisation contributions to uncertainty  $u_{char}$  are in the range of 0.2%.

### Traceability:

All certified values of Certipur® AAS single element standards are traceable to the corresponding NIST Standard Reference Materials (SRM) via an unbroken chain of calibrations.

### Uncertainty evaluation:

The expanded uncertainty  $U_{CRM}$  reported with the certified values is calculated in accordance to GUM and EA-4/02, with  $k=2$  as the coverage factor for a 95% coverage probability. Uncertainty contributions to the certified expanded uncertainty are illustrated by the following cause-and-effect-diagram (Ishikawa-Diagram):



The expanded uncertainty  $u_{CRM}$  is obtained from the standard uncertainties of characterisation, homogeneity and stability:

$$u_{CRM} = k \cdot u_{CRM}$$

$$u_{CRM} = \sqrt{u^2_{\text{Characterisation}} + u^2_{\text{Homogeneity}} + u^2_{\text{Stability}}}$$

$$u^2_{\text{Stability}} = u^2_{\text{sts}} + u^2_{\text{Its}}$$

Quality management system:

Certipur® AAS single element standards have been prepared and certified under an ISO 9001 quality management system in accordance to

ISO 17034: General requirements for the competence of reference material producers

ISO Guide 35: Reference materials – Guidance for characterization and assessment of homogeneity and stability

ISO Guide 31: Reference materials – Contents of certificates, labels and accompanying documentation

Eurachem / CITAC Guide: Quantifying uncertainty in analytical measurement Guide to the Expression of Uncertainty in measurement (GUM)

DIN EN ISO / IEC 17025: General requirements for the competence of testing and calibration laboratories

