Certification Report – Certified Reference Material

Certipur® Volumetric 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® Volumetric standards.

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

Preparation and packaging of CRMs:

Certipur® Volumetric standards are prepared from high purity salts. The mass fraction is measured by means of potentiometric titration.

Certipur® Volumetric standards are delivered in glas bottles. The label on the products is in accordance to ISO Guide 31 and covers ordering 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 with 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-unit homogeneity. Measurements have been carried out according to DIN EN ISO/IEC 17025. Results from multiple samples of the chosen bottles were evaluated.

Typical homogeneity contributions to uncertainty u_{hom} are in the range of 0.03%.

An evaluation of within-bottle (within-unit) homogeneity was conducted for assessment of the minimum sample size and does therefore not contribute to the uncertainty budget of this certified reference material. The minimum weigh-in quantity depends on the volumetric standard and is mentioned on the certificate of analysis.

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 (transportation) 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 according to DIN EN ISO/IEC 17025. Results were evaluated using linear regression and ANOVA and the calculated uncertainty 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 0.02%.

Short-term (transportation) stability:
Short-term (transportation) stability was evaluated using the classical experimental layout. Samples were stored at elevated temperatures that may arise during shipment of the products before measurement. Results were evaluated using linear regression and ANOVA and the calculated uncertainty included into the uncertainty budget as short- term (transportation) stability contribution u_{sts} .

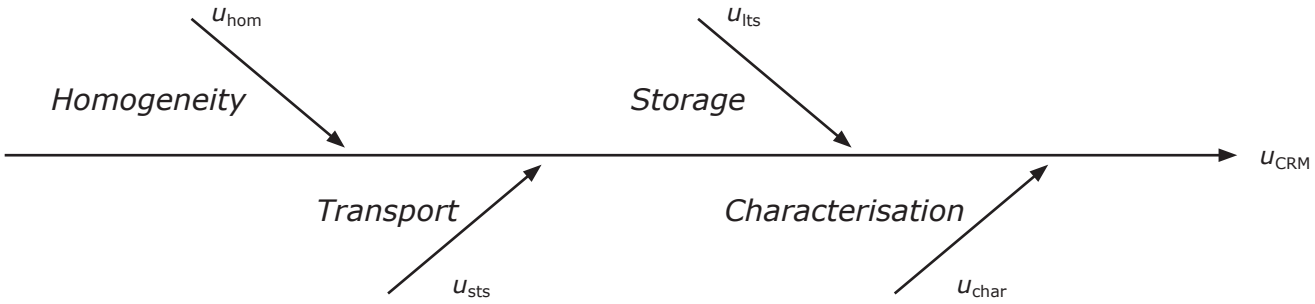
Typical short-term (transportation) stability contributions to uncertainty usts are in the range of 0.01%.

Stability monitoring:
Merck KGaA, Darmstadt, Germany will monitor all Certipur® Volumetric 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® Volumetric standards is carried out by the accredited quality control (QC) laboratory at Merck KGaA, Darmstadt, Germany according to DIN EN ISO / IEC 17025 by measuring the mass fraction by potentiometric titration. Typical characterisation contributions to uncertainty u_{char} are in the range of 0.010 – 0.025% depending on the volumetric standard.

Traceability:
All certified values of Certipur® Volumetric standards are directly traceable to the corresponding / suitable primary standard from NIST (NIST: National Institute of Standards and Technology).

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):



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

$$\boldsymbol{U}_{CRM} = k \cdot \boldsymbol{u}_{CRM}$$

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

$$\boldsymbol{u}^2\text{Stability} = \boldsymbol{u}^2\text{sts} + \boldsymbol{u}^2\text{lts}$$

Quality management system:
Certipur® Volumetric 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