

Traceability

The certified value "Purity" is determined by quantitative NMR (qNMR). Quantification by qNMR is traceable to NIST (using NIST SRM 350b (Benzoic acid) or other NIST compounds). The certified value "Identity" is traceable via cas number to the mol. The balances used are calibrated with weights traceable to the national standards (DKD). This makes the values traceable to the SI.

CRM solutions are traceable to the neat CRM by batch number. The certified value "Concentration" is traceable via the balances that were used in the preparation of the solutions. The balances are calibrated with weights traceable to the national standards (DKD).

Uncertainty Calculation

The reported uncertainty U is an expanded uncertainty according to EURACHEM / CITAC guide CG4 – Quantifying Uncertainty in Analytical Measurement – third edition.[1] The following scheme displays the factors contributing to measurement uncertainties:

For neat CRM, the combined uncertainty u_c is calculated by the combination of the uncertainty factors $u(\text{Homogeneity})$ and $u(\text{Assay Characterization})$ as well as $u(\text{short-term stability})$ and $u(\text{long-term stability})$. The reported uncertainty U that is given in the Certificate of Analysis of the Certified Reference Material is calculated $U = k \cdot u_c$ using a coverage factor of $k = 2$, which gives a level of confidence of 95%.

Homogeneity

The homogeneity testing was performed using random replicate samples of the final packaged CRM. If the batch is in accordance with the previous batch regarding purity and preparation then the homogeneity is performed by picking random samples from the batch. The samples have been thoroughly analyzed to prove homogeneity in accordance with internal procedures. The homogeneity is confirmed by ANOVA evaluation or similar procedures consistent with ISO 17034:2017. The same procedures apply for the determination of the uncertainty $u(\text{homogeneity})$.

Assay Characterization

The purity of the CRM and its uncertainty are determined by qNMR. The results of the qNMR measurement are compared with the results of other methods (chromatography, titration, etc.). The purity that was determined via qNMR is validated if it is not significantly different from the purity determined by other methods.

Stability

The stability of this product is based upon short term and long term studies evaluating the effect of temperature and packing on the product.

Short-Term Stability

Short-term storage conditions are intensely studied to verify the stability of the product during transport. The product is stored for a minimum of 3 days at 50 ± 3 °C and thoroughly analyzed according to internal procedures. Products are only shipped if they have been proven to be stable for the shipping conditions. The uncertainty of short-term stability is determined from different factors, including the uncertainty of the linear regression. The uncertainty of short-term stability is considered in the expanded uncertainty of the product.

Long-Term Stability

Long-term storage stability is verified by applying the recommended storage conditions given on the Certificate of Analysis. The product is analyzed in appropriate intervals using internal procedures. Long-Term stability monitoring was performed one year prior to shipping. If stability data of at least one year is available of former batches of neat products or their solutions, this data is used to determine the expiry date. Stability monitoring will be ongoing as well. If measurement values show a significant reduction in product stability, the storage conditions given on the Certificate of Analysis will be adjusted. The customers will be informed immediately. The uncertainty of long-term stability is determined from different factors including the uncertainty of the linear regression. The uncertainty of long-term stability is considered in the expanded uncertainty of the product.

References

[1]

S L R Ellison and A Williams (Eds). Eurachem/CITAC guide: Quantifying Uncertainty in Analytical Measurement, Third edition, (2012) ISBN 978-0-948926-30-3. Available from www.eurachem.org.

Version	Reason for Change	Date
1	Initial Version	01 Nov 2021