TraceCERT®
Traceable Certified Reference Materials

SIGMA-ALDRICH®

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: **Multi Anion Standard 2 for IC**

Product No.: **53798**

Lot: **BCBR6363V**

Composition: High-purity starting materials in in high-purity water (18.2 MΩ·cm, 0.22 µm filtered).

Density at 20°C: ρ = 1002.7 kg m^{-3} $u_c(\rho)$ = 0.5 kg m^{-3}

Intended use: Calibration of ion chromatography or any other analytical technique.

Storing and handling: This reference material shall be stored in the original closed bag between 5 °C and 30 °C. Before every use of the material the bottle must be shaken well and its temperature has to be 20°C. 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: **MAR 2020** (unopened bottle in aluminized bag)

Certificate issue date: 12 MAY 2016

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 values at 20°C and expanded uncertainty [$U = k \cdot u_c$; k = 2]	
Bromide (Br^-)	**997 mg kg^{-1} ± 3 mg kg^{-1}**	**1'000 mg L^{-1} ± 3 mg L^{-1}**
Chloride (Cl^-)	**996 mg kg^{-1} ± 3 mg kg^{-1}**	**999 mg L^{-1} ± 3 mg L^{-1}**
Fluoride (F^-)	**997 mg kg^{-1} ± 3 mg kg^{-1}**	**1'000 mg L^{-1} ± 3 mg L^{-1}**

1. Concept of Certification and Traceability Statement

To guarantee top reliability of the values for this *Trace***CERT**® certified reference material two independent procedures were followed. The values have to agree in the range of their uncertainties, but the value from the gravimetric preparation has been chosen as certified value [4]:

1. Gravimetric preparation using pure materials is a practical realization of concentration units, through conversion of mass to amount of substance [4]. 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 *Trace***CERT**® 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 (e.g. NIST or BAM) 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.

Sigma-Aldrich Production GmbH, Industriestrasse 25, 9471 Buchs/Switzerland,
Tel +41-81-755-2511, Fax +41-81-756-5449

2. Content 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.

3. Traceability Measurements

Only internationally accepted reference materials e.g. from NIST (USA) or BAM (Germany) have been carefully selected to provide the basis for traceability to the SI unit Mole. 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:

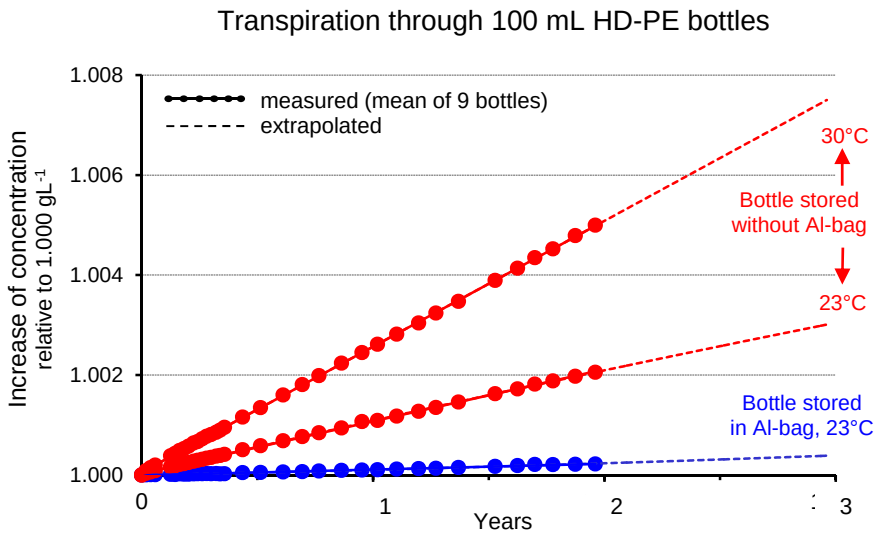
Constituent	Starting material	Reference	Method
Bromide (Br^-)	NaBr	NIST SRM 999	Argentometric titration
Chloride (Cl^-)	NaCl	NIST SRM 999	Argentometric titration
Fluoride (F^-)	NaF	NIST SRM 3183	Ion chromatography

4. Storing Behavior

The storage behavior of standard solutions is of greatest importance with regard to the certified value. Therefore the two most important effects were investigated by in-depth studies in a cooperation with EMPA, St. Gallen:

1. The leach out of trace impurities from HDPE (high-density polyethylene) bottles was determined with HR-ICP-MS after leaching the bottles with 2% nitric acid. Maximum contamination levels were found in the ng L^{-1} level for 12 elements.

2. To avoid significant loss of mass through transpiration the bottle is delivered in aluminum coated bags. After the bottle has been removed from the bag, transpiration will occur at an accelerated rate (see figure). We highly recommend not to open the bag until the solution is needed. Once the bottle is opened the solution should be stored at reduced temperature (4°C) to reduce transpiration.



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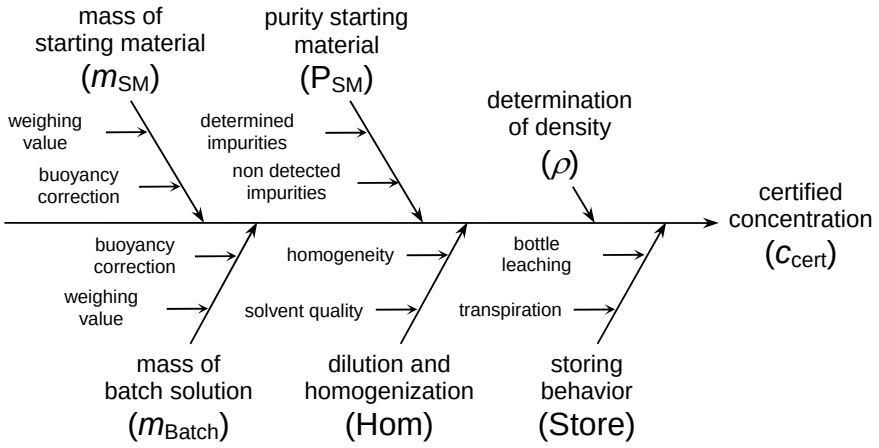
5. UNCERTAINTY EVALUATION

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 [5]:



Typical contributions:

$u(m_{SM})$	< 0.01%
$u(m_{Batch})$	< 0.01%
$u(P_{SM})$	< 0.1%
u(Hom)	< 0.03%
u(Store)	< 0.09%
$u(\rho)$	< 0.05%

Combined uncertainty [6]:

$u_c(c_{cert})$ < 0.2%

Expanded uncertainty:

$U(c_{cert})$ < 0.3%

CRM operations: S.Matt

S.Matt

Certification body: Klaus Dietz Schmidt

K.-D. Schmidt, Ph.D.

ISO Guide 34 **ISO/IEC 17025** **ISO 9001**

[1] ISO Guide 31:2000, "Reference materials - Contents of certificates and labels"
[2] ISO Guide 35:2006, "Reference materials - General and statistical principles for certification"
[3] Eurachem/CITAC Guide, 3rd Ed. (2012), "Quantifying uncertainty in analytical measurement"
[4] Eurachem/CITAC Guide, 1st Ed. (2003) "Traceability in chemical measurement"
[5] 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.
[6] Calculated by combination of the squared contribution values

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