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: **Chlorate standard for IC**

Product No.: **73166** Lot: **BCBR4341V**

Composition: Potassium chlorate (pure material) in high-purity water (18.2 MΩ·cm, 0.22 µm filtered).

Density at 20°C: ρ = 999 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 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: **JAN 2019** Certificate issue date: 28 JAN 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 value at 20°C and expanded uncertainty [$U = k \cdot u_c$; k = 2]	
Chlorate	**1'001 mg kg^{-1} ± 4 mg kg^{-1}**	**1'000 mg L^{-1} ± 4 mg L^{-1}**

1. Concept of Certification and Traceability Statement

To guarantee top reliability of the values for this *Trace***CERT**® 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 [4]:

1. Gravimetric preparation using pure materials is a practical realization of concentration units, through conversion of masses and mole fraction to mass fraction [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 highest purity are used (see paragraph 2). All balances are certified by DKD and calibrated with OIML Class E2 (up to 12 kg) and F2 (up to 64 kg) weights. The bulk solution was homogenized by overhead tumbling in a PVDF container for at least 6 hours.
2. The starting material is measured against a certified reference material (i.e. 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.
3. Whenever applicable the bottled *Trace***CERT**® calibration solution is compared to a second reference 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.

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3. Traceability Measurements

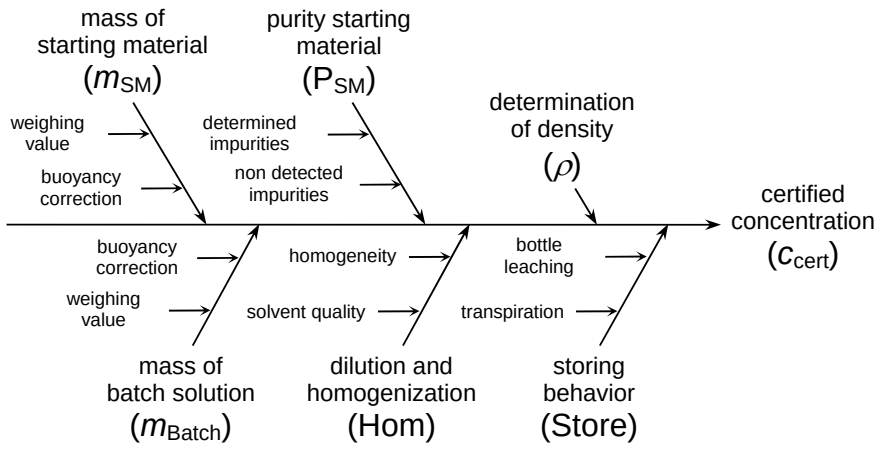
Only internationally accepted reference materials e.g. from NIST (USA) or BAM (Germany) have been carefully selected to provide the basis for traceability to the SI unit mole. When no such reference is available, an elemental metal or an adequate salt of highest available purity is used to confirm traceability 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:	Sodium oxalate, certified by BAM (Sigma-Aldrich No. 71804) / redox titration
bottled solution:	Sodium chloride, certified by BAM (Sigma-Aldrich No. 71387) / argentometric titration

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 [5]:



Typical contributions:

$u(m_{SM})$	< 0.01%
$u(m_{Batch})$	< 0.01%
$u(P_{SM})$	< 0.05%
$u(Hom)$	< 0.03%
$u(Store)$	< 0.20%
$u(\rho)$	< 0.05%

Combined uncertainty [6]:

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

Expanded uncertainty:

$\boldsymbol{U(c_{cert})}$	**< 0.4%**

5. Trace Impurities in Bottled Solution

The following anions were measured as possible impurities (in µg kg^{-1}, <X = below detection limit, m = matrix)

Bromate	Bromide	Chloride	Chlorite	Fluoride	Iodide	Nitrite	Nitrate	Sulfate	Phosphate
< 25	< 25	< 25	< 25	< 25	< 25	< 25	< 25	< 25	< 25

CRM operations: S.Matt

S.Matt

Certification body: Klaus Dieter 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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