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:	**Tartrate standard for IC**
Product no.:	**43484**
Lot no.	**BCBR1830V**
Composition:	Tartaric acid (high purity quality) in high-purity water (18.2 MΩ·cm, 0.22 µm filtered). The bottled solution is stabilized with sodium azide (about 5 mg/L) and additionally filtered through a 0.2 µm membrane.
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:	09 MAR 2016
Bottle opening date:	

The certified values and uncertainties are according to ISO Guide 35 [2] and Eurachem/CITAC Guide [3]

Constituent	Certified value at 20°C and expanded uncertainty [$U = k \cdot u_c$; $k = 2$]	
Tartrate	**1’001 mg kg^{-1} ± 5 mg kg^{-1}**	**1’000 mg L^{-1} ± 5 mg L^{-1}**
Traceability [4]	NIST SRM 84l, Potassium Hydrogen Phthalate	

Measurand	Certified value and expanded uncertainty [$U = k \cdot u_c$; $k = 2$]
Density at 20°C	**0.9988 g mL^{-1} ± 0.0005 g mL^{-1}**

CRM operations: S. Matt
S.Matt

Certification body:
K.-D. Schmidt, Ph.D.

ISO Guide 34 ISO/IEC 17025 ISO 9001

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

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1. Concept of Certification

The certified concentrations and expanded uncertainties of the analyte are based on the results obtained from gravimetrical production and from the analytical results determined using high-resolution quantitative NMR, which is recognized as primary measurement method.

Gravimetric preparation using well defined and pure materials is a practical realization of concentration units, through conversion of mass to amount of substance [4]. All high-precision balances are periodically calibrated by a third party and certified according to DAkkS guidelines (DAkkS = Deutsche Akkreditierungsstelle GmbH, which is the national accreditation body for the Federal Republic of Germany).

Production and certification of this CRM are performed under double-accreditation in accordance with ISO/IEC 17025 [5] and also ISO Guide 34 [6]. Storage stability, leaching and homogeneity tests are also considered for certification.

2. Starting Material Content by qNMR

The absolute content of starting material is measured by high-resolution quantitative NMR measurements on a Bruker 600 MHz Avance III NMR spectrometer.

The certification of the content is performed using 5-10 separate samples which are each spiked with an adequate amount of internal reference and then immediately dissolved in deuterated solvent. In most cases 16-32 scans are recorded for every sample with a ^{1}H relaxation time of d1 = 60 seconds. Quantification of the content is directly calculated from the ^{1}H-NMR peak areas and the initial weights of the sample and reference substance. After ANOVA the resulting standard deviation is included into the uncertainty calculation of the certified value.

Extensive stability and homogeneity tests are considered for certification.

Accelerated stability test is performed with samples which are stored above the recommended storage temperature. The material is tested after 1, 3, 9 and 18 months.

Long term stability test is performed with samples which are stored at the recommended storage temperature and qNMR double determination after 24 and 48 months.

Homogeneity of the material is tested by qNMR measurements using 5-10 subsamples which are taken from different positions in the entire bulk material. The recommended minimal sample size is taken for all the homogeneity test samples. Analysis of variance (ANOVA) results are included into the calculation of content uncertainty of this CRM.

Starting material	Starting material content with expanded uncertainty	Traceable to
L-(+)-Tartaric acid P/N 41447 Lot BCBM2140V	**99.9 % ± 0.3 %**	NIST SRM 84l, potassium hydrogen phthalate and NIST SRM 350b, benzoic acid

3. Density Measurement

The density measurement is carried out according to ISO 15212-1 [7] and using the digital density meter DMA 4500M from Anton Paar with an oscillating U-tube installed. The measurement uncertainty is calculated according to Eurachem/CITAC Guide and reported as combined expanded uncertainty at the 95% confidence level.

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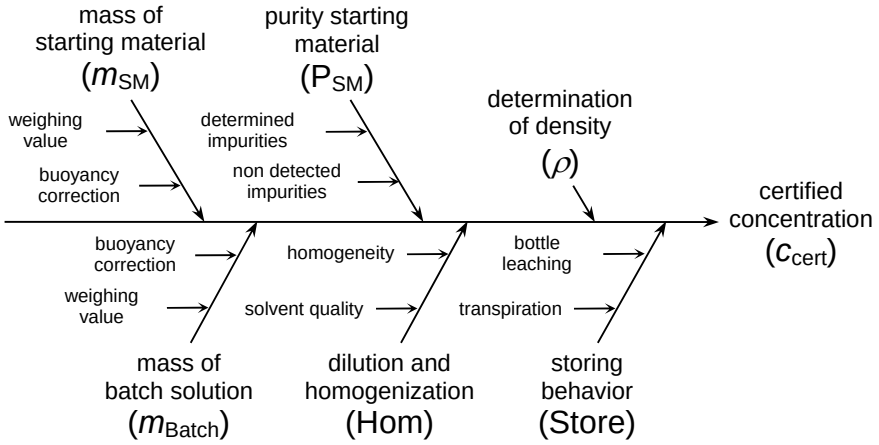
4. UNCERTAINTY EVALUATION

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 [8]:



Typical contributions:

$u(m_{SM})$	< 0.01%
$u(m_{Batch})$	< 0.01%
$u(P_{SM})$	< 0.15%
u(Hom)	< 0.03%
u(Store)	< 0.20%
$u(\rho)$	< 0.05%

Combined uncertainty [9]:

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

Expanded uncertainty:

$\boldsymbol{U(c_{cert})}$ < **0.5%**

References

[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] ISO/IEC 17025, 2nd Ed. (2005), “General requirements for the competence of testing and calibration laboratories”
[6] ISO Guide 34:2009, “General requirements for the competence of reference material producers”
[7] DIN EN ISO 15212-1:1998, Oscillation-type density meters - Part 1: Laboratory instruments
[8] 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.
[9] Calculated by combination of the squared contribution values

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