

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: **Lactose Standard for IC**

Product no.: **72622**

Lot no.: **BCBQ6479V**

Composition: Lactose (pure material) in water **TraceSELECT**[®]Ultra (18.2 MΩ·cm, 0.22 μm filtered). The HDPE bottles are x-ray sterilized and the solution is additionally filtered through a 0.2 μm membrane and bottled under clean room conditions.

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

Storing and handling: This reference material shall be stored in the dark at reduced temperature (e.g. refrigerator) whereas the certified value is guaranteed when the long-term storage temperature is between 2 to 5°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.

Expiry date: **AUG 2017**

Traceability^[2]: Traceable to SI unit kg. For further details see section 2.

Certificate issue date: 23 SEPT 2015

The certified values and uncertainties are according to ISO Guide 35^[3] and Eurachem/CITAC Guide^[4].

Constituent	Certified values at 20°C and expanded uncertainties [$U = k \cdot u_c$; $k = 2$]	
Lactose (C ₁₂ H ₂₂ O ₁₁)	999 mg L⁻¹ ± 6 mg L⁻¹	1000 mg kg⁻¹ ± 6 mg kg⁻¹

Measurand	Certified value and expanded uncertainty [$U = k \cdot u_c$; $k = 2$]
Density at 20°C	0.9986 g mL⁻¹ ± 0.0005 g mL⁻¹

CRM operations:

H. Sprecher

H.Sprecher

Certification body:

Klaus-D. Schmidt

K.-D. Schmidt, Ph.D.



ISO Guide 34

ISO/IEC 17025

ISO 9001

1. CONCEPT OF CERTIFICATION

The certified concentrations and expanded uncertainties of the analyte is based on the results obtained from gravimetric production and from the analytical results determined using mass balance approach.

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 MASS BALANCE

For pure materials ($P > 99\%$) the most appropriate way of purity determination is to quantify the impurities (w_i) and to subtract the sum from 100%.

$$P = 100\% - \sum_i w_{i, \text{found}}$$

Starting material	Starting material content with expanded uncertainty	Traceable to
Lactose P/N PHR1025 Lot P500190	99.3 % ± 0.5 %	SI unit kg by mass balance

The methods applied for the determination of impurities are High-performance liquid chromatography, GC-MS headspace, Karl-Fischer titration and gravimetric analysis.

Homogeneity of the starting material was assessed in accordance with ISO Guide 35. Completed units were sampled using a random stratified sampling protocol. The results of chemical analysis were then compared by Single Factor Analysis of Variance (ANOVA). The uncertainty due to homogeneity was derived from the ANOVA. Heterogeneity was not detected under the conditions of the ANOVA.

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.

4. UNCERTAINTY EVALUATION

All uncertainties are calculated according to Eurachem/CITAC Guide and reported as combined expanded uncertainties at the 95% confidence level. The main uncertainty contributions are illustrated by the following cause-effect diagram^[8].

Typical relative contributions are:

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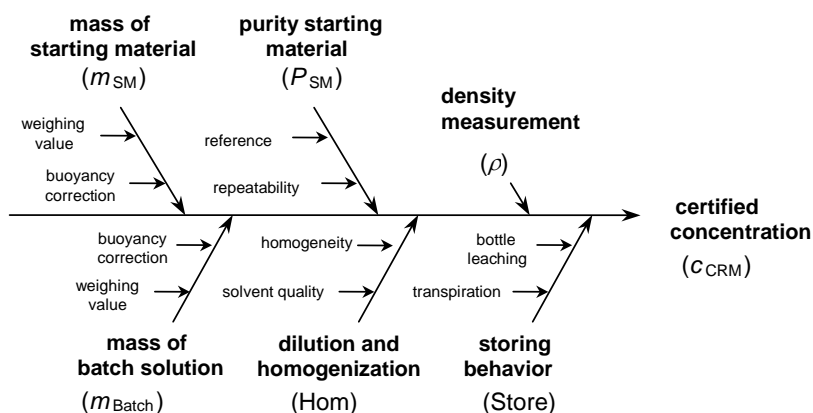
$u(m_{SM})$	< 0.01 %
$u(m_{Batch})$	< 0.01 %
$u(P_{SM})$	< 0.25 %
$u(Hom)$	< 0.01 %
$u(Store)$	< 0.14 %
$u(\rho)$	< 0.05 %

Combined uncertainty^[9]:

$u_c(c_{cert})$	< 0.3 %
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Expanded uncertainty:

$U(c_{cert})$	< 0.6 %
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The combined uncertainty u_c is calculated by combination of the squared contribution values. The expanded uncertainty U is then calculated to a confidence level of 95%, by multiplying u_c with a confidence level factor of $k=2$.

References

- [1] ISO Guide 31, 2nd Ed. (2000), "Reference materials - Contents of certificates and labels"
- [2] Eurachem/CITAC Guide, 1st Ed. (2003), "Traceability in chemical measurement"
- [3] ISO Guide 35, 3rd Ed. (2006), "Reference materials - General and statistical principles for certification"
- [4] Eurachem/CITAC Guide, 2nd Ed. (2000), "Quantifying uncertainty in analytical measurement"
- [5] ISO/IEC 17025, 2nd Ed. (2005), "General requirements for the competence of testing and calibration laboratories"
- [6] ISO Guide 34, 3rd Ed. (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