

Certified Reference Material Reference material certificate

Glycolate Standard for IC

TraceCERT®
 Traceable Certified Reference Materials

Product no.: 07391
Lot no.: BCCJ6115
Description of CRM: Glycolic acid (pure material) 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.
Expiry date: OCT 2025
Storage: Store at 5°C-25°C
Density (certified) at 20°C: 998.6 kg m⁻³ ± 0.5 kg m⁻³

Constituent Certified values at 20°C and expanded uncertainties, $U = k \cdot u$ ($k = 2$) ^{[1][2]}

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Glycolate	1001 mg kg ⁻¹ ± 4 mg kg ⁻¹ 1000 mg L ⁻¹ ± 4 mg L ⁻¹

Metrological traceability: NIST SRM 84I, potassium hydrogen phthalate
 Details see "Details on metrological traceability".^[3]
Measurement method: The certified value is determined by high-precision weighing of starting materials characterized by quantitative NMR.
Intended use: Calibration of ion chromatography or any other analytical technique.
Instructions for handling and correct use: The bottle's temperature must be 20°C. Shake well before every use. If storage of a partially used bottle is necessary (at the user's risk), the cap should be tightly sealed and the bottle should be stored at reduced temperature (e.g. refrigerator) to minimize transpiration rate.
Health and safety information: Please refer to the Safety Data Sheet for detailed information about the nature of any hazard and appropriate precautions to be taken.
Packaging: 100 mL HDPE bottle
Accreditation: Sigma-Aldrich Production GmbH is accredited by the Swiss Accreditation Service SAS as reference material producer under no. SRMS 0001 in accordance with international standard ISO 17034^[4]
Certificate issue date: 22 NOV 2022


 ISO 17034
 SRMS 0001

S. Matt – CRM Operations

Dr. P. Zell – Approving Officer



Certification process details:

The certified concentrations and expanded uncertainties of the analyte are based on the results obtained from gravimetric 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 ^[3]. 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 in accordance with **ISO 17034** ^[4]. Storage stability, leaching and homogeneity tests are also considered for certification.

Details on metrological traceability:

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 ¹H relaxation time of d1 = 60 seconds. Quantification of the content is directly calculated from the ¹H-NMR peak areas and the initial weights of the sample and reference substance. After Analysis of variance (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 storing temperature (mostly at 45 °C) and qNMR double determinations after 1, 3, 9 and 18 months.

Long term stability test is performed with samples which are stored at the recommended storing 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. ANOVA results are included into the calculation of content uncertainty of this CRM.

Starting material	Starting material content with expanded uncertainty	Traceable to
Glycolic acid P/N 94815 Lot BCCD6978	99.9% +/- 0.2%	NIST SRM 84I, potassium hydrogen phthalate

Homogeneity assessment:

Due to the production process, a homogeneous solution derives. Nevertheless a small homogeneity contribution is included into the calculation of content uncertainty of this CRM.

Stability assessment:

Two major contributors to the overall stability of this CRM were evaluated in deep. The mass loss due to evaporation is driven by the storage temperature and the geometry and the material of the bottle used. The bottles were tested for long term weight loss due to evaporation at different temperatures. The effect was taken into account in the uncertainty contribution of the storage stability. The microbiological stability was assessed with microbiological testing of the bottled solution. No biological contamination could be detected.

Density Measurement:

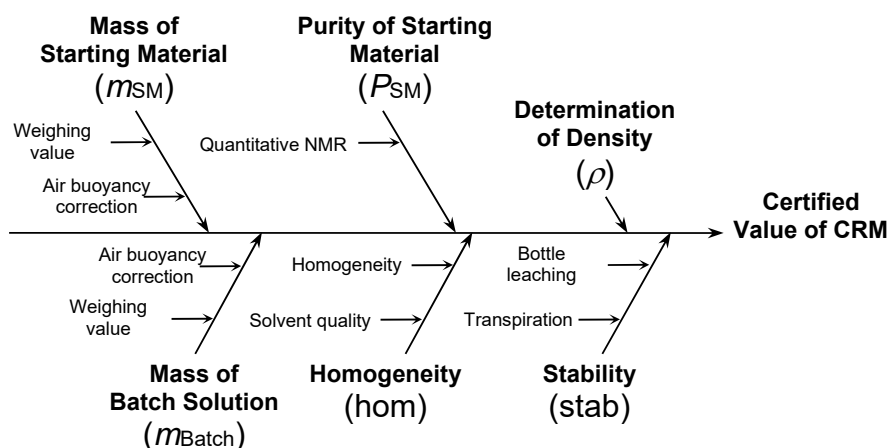
The density measurement is carried out under the scope of the ISO/IEC 17025 accreditation according to ISO 15212-1 ^[5] and using the digital density meter DMA 4500M from Anton Paar with an oscillating U-tube installed. The measurement uncertainty is calculated according to Eurachem/CITAC Guide and reported as combined expanded uncertainty at the 95% confidence level, using a coverage factor of $k = 2$.

Uncertainty evaluation:

The uncertainty contributions are illustrated by the following cause-effect diagram [6]:

Typical relative contributions are:

$u(m_{SM})$	< 0.01 %
$u(m_{Batch})$	< 0.01 %
$u(P_{SM})$	< 0.10 %
u_{hom}	< 0.03 %
u_{stab}	< 0.20 %
$u(\rho)$	< 0.05 %



The combined standard uncertainty is calculated by combination of the standard uncertainties of the input estimates according to Eurachem/CITAC Guide "Quantifying Uncertainty in Analytical Measurement" and ISO 17034.[2][4]

Expanded uncertainty is then calculated to a confidence level of 95%, typically by multiplying with a confidence level factor of $k=2$.

References:

- [1] ISO Guide 35:2017, "Reference materials - Guidance for characterization and assessment of homogeneity and stability"
- [2] Eurachem/CITAC Guide, 3rd Ed. (2012), "Quantifying uncertainty in analytical measurement"
- [3] Eurachem/CITAC Guide, 2nd Ed. (2019), "Metrological Traceability in chemical measurement"
- [4] ISO 17034:2016, "General requirements for the competence of reference material producers"
- [5] DIN EN ISO 15212-1:1998, Oscillation-type density meters - Part 1: Laboratory instruments
- [6] 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.

Certificate of analysis revision history:

Certificate version	Certificate issue date	Reason for version
01	22 NOV 2022	Initial version

Disclaimer:

The purchaser must determine the suitability of this product for its particular use. Sigma-Aldrich Production GmbH makes no warranty of any kind, express or implied, other than its products meet all quality control standards set by Sigma-Aldrich Production GmbH. We do not guarantee that the product can be used for a special application.

