Certificate of Analysis – Certified Reference Material

Heavy metal mix IX

TraceCERT®
Traceable Certified Reference Materials

Product no.: 89471
Lot no.: BCCD3328
Description of CRM: High-purity starting materials in 12% HNO_3 (prepared with high purity water, 18.2 MΩ·cm, 0.22 µm filtered, and acid suitable for trace analysis)
Expiry date: JUN 2024
Storage: Store at 5°C-25°C
Density (certified) at 20°C: 1.0656 g · cm^{-3} ; $u(\rho)$ = 0.0005 g · cm^{-3}

Constituent	Certified values at 20°C and expanded uncertainties, $U = k \cdot u$ (k = 2) [1][2]	
Arsenic (As)	**90.1 mg/kg ± 0.3 mg/kg**	**96.0 mg/l ± 0.3 mg/l**
Cadmium (Cd)	**90.1 mg/kg ± 0.3 mg/kg**	**96.0 mg/l ± 0.3 mg/l**
Lead (Pb)	**90.1 mg/kg ± 0.3 mg/kg**	**96.0 mg/l ± 0.3 mg/l**
Mercury (Hg)	**90.0 mg/kg ± 0.3 mg/kg**	**95.9 mg/l ± 0.3 mg/l**

Metrological traceability: Traceable to the SI unit kg via starting materials characterization using NIST SRMs. Details see "Details on metrological traceability" on page 2. [3]

Measurement method: The certified value is determined by high-precision weighing of thoroughly characterized starting materials and verified by measurement against NIST SRMs.

Intended use: Calibration of ICP, AAS, spectrophotometry 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.

Accreditation: Sigma-Aldrich Production GmbH is accredited by the Swiss accreditation authority SAS as registered reference material producer SRMS 0001 in accordance with ISO 17034 and registered testing laboratory STS 0490 according to ISO/IEC 17025. [4][5]

Certificate issue date: July 29, 2020

ISO 17034 SRMS 0001 | **ISO/IEC 17025 STS 0490** | **ISO 9001 005356 QM08**

H.Sprecher– CRM Operations

Dr. P. Zell – Approving Officer

Sigma-Aldrich Production GmbH, Industriestrasse 25, 9471 Buchs, Switzerland;
Tel +41-81-755-2511; Fax +41-81-756-5449; www.sigmaaldrich.com
Sigma-Aldrich Production GmbH is a subsidiary of Merck KGaA, Darmstadt, Germany.

Packaging: 100 mL FEP bottle, sealed with an aluminium bag

Certification process details:

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 [3]:

1. Gravimetric preparation using pure materials is a practical realization of concentration units, through conversion of mass to amount of substance [3]. 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 from NIST 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. Density measurement is performed under accreditiation to ISO/IEC 17025.

Details on metrological traceability:

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
Arsenic (As)	As_2O_3	NIST SRM 83	Redox titration
Cadmium (Cd)	Cd metal	NIST SRM 728	Complexometric titration
Lead (Pb)	$Pb(NO_3)_2$	NIST SRM 728	Complexometric titration
Mercury (Hg)	Hg metal	NIST SRM 728	Complexometric titration

Details on 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.

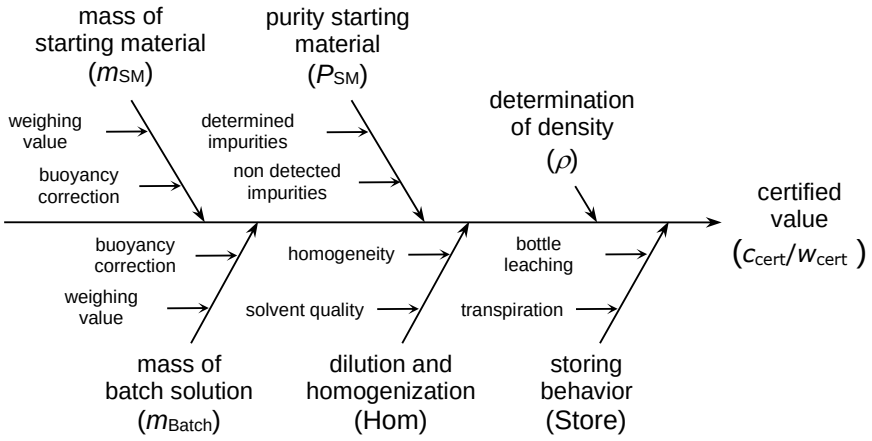
Homogeneity assessment:
Due to the production process, a homogeneous solution derives. Nevertheless a small homogeneity contribution is included into the calculation of content uncertainty of this CRM.

Stability assessment:
The storage behavior of standard solutions is an important factor regarding the certified value. An ideal container for a CRM solution is chemically inert, completely tight and does neither adsorb the analyte nor leach trace impurities into the solution. Therefore the most important storing effects were investigated by in-depth studies.
Every plastic bottle (e.g. PP, HDPE, FEP, PFA) is untight for a certain range due to the transpiration of solvent through the wall which leads to an increase of the analyte concentration over time. To avoid significant loss of solvent through Transpiration, the bottle is delivered in aluminum coated bags. When the CRM is in the sealed Al-bag, the change in concentration at 23°C is less than 0.02% per year. After the bottle has been removed from the bag, transpiration will occur at an accelerated rate depending on the temperature. 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 minimize transpiration rate.

Associated uncertainty:
All uncertainties are calculated according to Eurachem/CITAC Guide [2] and reported as combined expanded uncertaintiesat the 95% confidence level. For gravimetric preparation 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(Store)	< 0.09 %
$u(\rho)$	< 0.05 %



Combined uncertainty [7]:
$u_c(c_{cert}/w_{cert})$ < 0.15 %

Expanded uncertainty:
$\boldsymbol{U(c_{cert}/w_{cert})}$ **< 0.3%**

Density Measurement:
The density measurement is carried out under the scope of the ISO/IEC 17025 accreditation according to ISO 15212-1[8] 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.

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, 1st Ed. (2003), "Traceability in chemical measurement"
[4] ISO 17034:2016, "General requirements for the competence of reference material producers"
[5] ISO/IEC 17025:2005, "General requirements for the competence of testing and calibration laboratories"
[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.
[7] Calculated by combination of the squared contribution values
[8] DIN EN ISO 15212-1:1998, Oscillation-type density meters - Part 1: Laboratory instruments

Certificate of analysis revision history:

Certificate version	Date	Reason for version
01	July 29, 2020	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.