Certified Reference Material
Reference material certificate

Chromium(VI) Standard for ICP

TraceCERT® Traceable Certified Reference Materials

Product no.:	19036
Lot no.:	BCCM1049
Description of CRM:	Ammonium dichromate (high-purity quality) in high-purity water (18.2 MΩ·cm, 0.22 µm filtered).
Expiry date:	JUL 2028 (unopened bottle in aluminized bag)
Storage:	Store at 5°C-25°C
Density (certified) at 20°C:	999.7 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]	
Chromium(VI)	**1000 mg kg^{-1} ± 2 mg kg^{-1}**	**1000 mg L^{-1} ± 2 mg L^{-1}**

Metrological traceability:	Certified values are traceable to the International System of units (SI) through a metrologically valid weighing process. Details see "Details on metrological traceability".[3]
Measurement method:	The certified value is determined by high-precision weighing of thoroughly characterized starting materials and verified by measurement against NIST SRMs or similar CRMs in accordance with ISO/IEC 17025.[4]
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.
Packaging:	100 mL HDPE bottle sealed with an aluminized bag
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[5]
Certificate issue date:	24 OCT 2024

ISO 17034
SRMS 0001

S. Matt – CRM Operations

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Certification process details:

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 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 this *Trace***CERT®** reference material 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 calibrated annually by an ISO/IEC 17025 accredited laboratory and certified according to DKD guidelines. Calibration is checked daily with OIML Class E2 or F2 weights.
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.
4. The certified chromium (VI) mass fraction is calculated from total chromium minus measured chromium (III). When chromium (III) mass fraction is below detection limit only additional uncertainty contribution is added.

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. 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: NIST SRM 136f / redox titration

bottled solution: NIST SRM 136f / redox 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:

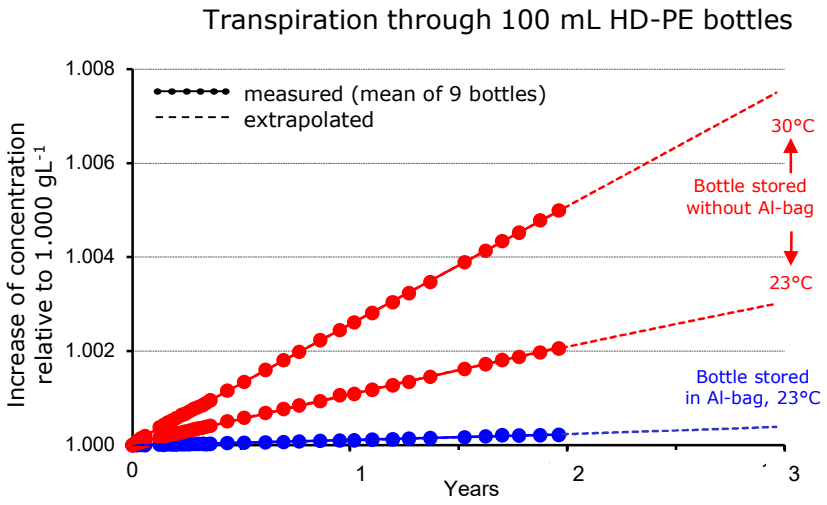
Due to the production process, a homogeneous solution derives. Nevertheless a small homogeneity contribution is included into the calculation of content uncertainty of this CRM.

Stability assessment:

The storage behavior of standard solutions is of greatest importance with regard to the certified value. Therefore the two most important effects were investigated by in-depth studies in a cooperation with EMPA, St. Gallen:

1. The leachate from HDPE (high-density polyethylene) bottles was analyzed by HR-ICP-MS after leaching the bottles with 2% nitric acid. Maximum contamination levels were found in the ng L^{-1} level for 12 elements.

2. To avoid significant loss of mass through transpiration the bottle is delivered in aluminum coated bags. After the bottle has been removed from the bag, transpiration will occur at an accelerated rate (see figure). We highly recommend not opening the bag until the solution is needed. Once the bottle is opened the solution should be stored at reduced temperature (4 °C) to reduce transpiration.



Density Measurement:

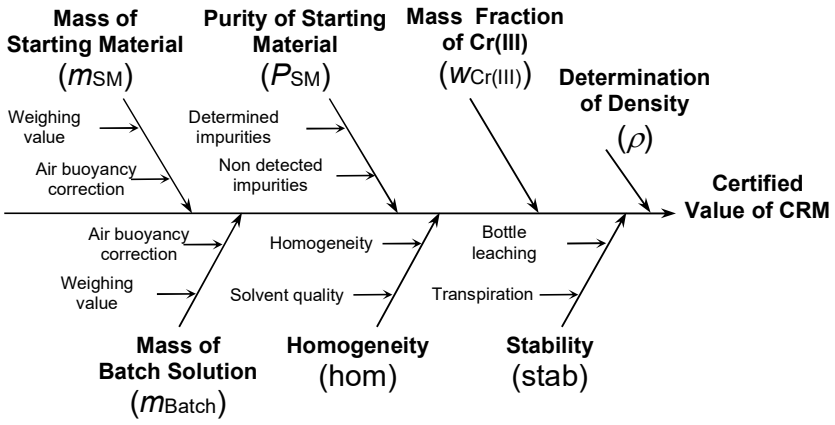
The density measurement is carried out in accordance with ISO/IEC 17025[4] and ISO 15212-1 [6] using the digital density meter DMA 4500M from Anton Paar with an oscillating U-tube installed. The measurement uncertainty is calculated according to Eurachem/CITAC Guide and reported as combined expanded 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 [7]:

Typical relative contributions are:

$u(m_{SM})$	< 0.01 %
$u(m_{Batch})$	< 0.01 %
$u(P_{SM})$	< 0.05 %
$U(w_{Cr(III)})$	< 0.02 %
u_{hom}	< 0.03 %
u_{stab}	< 0.09 %
$u(\rho)$	< 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][5]

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

Trace Impurities:

The chromium (III) ion species is determined in the bottled solution. Therefore the two chromium species were separated by suitable chromatography method and then measured with ICP-MS. Heightening experiments were conducted to verify the recovery rate and the reliability of the method.

Chromium (III) < 0.2 mg kg^{-1}

Up to 75 trace impurities were analyzed by ICP-OES, ICP-MS and AAS. Some of the impurities are determined in the starting material and calculated for the solution (e.g. for rare earth elements contamination during the preparation is rendered impossible). Other elements are determined both in the starting material as well as in the bottled solution.

All values listed below are given in mg kg^{-1} (ppm), <X = below detection limit, m = matrix, n.a. = not analyzed

Li	Be											B	C	N	O	F	Ne
<0.010	<0.005											<0.010	n.a.	n.a.	n.a.	n.a.	n.a.
Na	Mg											Al	Si	P	S	Cl	Ar
<0.10	<0.005											<0.010	<0.020	<0.002	<0.050	n.a.	n.a.
K	Ca	Sc	Ti	V	Cr	Mn	Fe	Co	Ni	Cu	Zn	Ga	Ge	As	Se	Br	Kr
<0.10	<0.010	<0.002	<0.010	<0.025	m	<0.010	<0.010	<0.010	<0.010	0.014	<0.010	<0.001	<0.050	<0.001	<0.002	n.a.	n.a.
Rb	Sr	Y	Zr	Nb	Mo	Tc	Ru	Rh	Pd	Ag	Cd	In	Sn	Sb	Te	I	Xe
<0.001	<0.010	<0.001	<0.010	<0.001	<0.050	n.a.	0.004	<0.001	<0.001	<0.001	<0.005	<0.001	<0.025	<0.001	<0.001	n.a.	n.a.
Cs	Ba	La	Hf	Ta	W	Re	Os	Ir	Pt	Au	Hg	Tl	Pb	Bi	Po	At	Rn
<0.001	<0.010	<0.001	<0.001	<0.001	<0.001	<0.001	<0.001	<0.001	<0.001	<0.001	<0.001	<0.50	<0.050	<0.25	n.a.	n.a.	n.a.
Fr	Ra	Ac															
n.a.	n.a.	n.a.															

Ce	Pr	Nd	Pm	Sm	Eu	Gd	Tb	Dy	Ho	Er	Tm	Yb	Lu
<0.001	<0.001	<0.001	n.a.	<0.001	<0.001	<0.001	<0.001	<0.001	<0.001	<0.001	<0.001	<0.001	<0.001

Th	Pa	U
<0.001	n.a.	<0.001

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] The accredited testing laboratory STS 0490 performs the measurements and weighing steps for the certification of this CRM under ISO/IEC 17025:2017, "General requirements for the competence of testing and calibration laboratories"
[5] ISO 17034:2016, "General requirements for the competence of reference material producers"
[6] DIN EN ISO 15212-1:1998, Oscillation-type density meters - Part 1: Laboratory instruments
[7] 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	24 OCT 2024	Initial version

Disclaimer:

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