**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: **Elemental Impurities according to ICH Q3D oral, Standard 2**

Product No.: **73108** Lot: **BCBV3650**

Composition: High-purity starting materials in 10% HCl (prepared with high purity water, 18.2 MΩ·cm, 0.22 µm filtered, and acid suitable for trace analysis).

Density at 20°C: $\rho$ = 1048.6 kg m$^{-3}$ $u_c(\rho)$ = 0.5 kg m$^{-3}$

Intended use: Calibration of ICP, AAS, spectrophotometry or any other analytical technique.

Storing and handling: This reference material shall be stored between 5 °C and 30 °C. The bottle's temperature must be 20°C and shaken well before every use. 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: **JUN 2021**

Certificate issue date: 04 SEP 2017

Certificate version: 01

Bottle opening date: _____

| Certified value traceable to SI unit kg and uncertainty according to ISO Guide 35 [2] and Eurachem/CITAC Guide [3] | | |
|---|---|---|
| Constituent | Certified values at 20°C and expanded uncertainty [$U = k \cdot u_c$; $k = 2$] | |
| **Gold (Au)** | **95.6 mg kg$^{-1}$ ± 0.5 mg kg$^{-1}$** | **100.2 mg L$^{-1}$ ± 0.6 mg L$^{-1}$** |
| **Iridium (Ir)** | **95.6 mg kg$^{-1}$ ± 0.8 mg kg$^{-1}$** | **100.2 mg L$^{-1}$ ± 0.9 mg L$^{-1}$** |
| **Osmium (Os)** | **95.6 mg kg$^{-1}$ ± 0.5 mg kg$^{-1}$** | **100.2 mg L$^{-1}$ ± 0.6 mg L$^{-1}$** |
| **Palladium (Pd)** | **95.6 mg kg$^{-1}$ ± 0.3 mg kg$^{-1}$** | **100.2 mg L$^{-1}$ ± 0.3 mg L$^{-1}$** |
| **Platinum (Pt)** | **95.6 mg kg$^{-1}$ ± 0.5 mg kg$^{-1}$** | **100.2 mg L$^{-1}$ ± 0.6 mg L$^{-1}$** |
| **Rhodium (Rh)** | **95.6 mg kg$^{-1}$ ± 0.4 mg kg$^{-1}$** | **100.2 mg L$^{-1}$ ± 0.5 mg L$^{-1}$** |
| **Ruthenium (Ru)** | **95.6 mg kg$^{-1}$ ± 0.8 mg kg$^{-1}$** | **100.2 mg L$^{-1}$ ± 0.9 mg L$^{-1}$** |

## 1. Concept of Certification and Traceability Statement

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 [4]:

1. Gravimetric preparation using pure materials is a practical realization of concentration units, through conversion of mass to amount of substance [4]. 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.



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

SIGMA-ALDRICH®

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.

## 2. Content of 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.

## 3. Traceability Measurements

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 |
|---|---|---|---|
| Gold (Au) | $H(AuCl_4)$ | NIST SRM 3121 | ICP-OES |
| Iridium (Ir) | $IrCl_3$ x 3 $H_2O$ | Traceable to in-house standard | ICP-OES |
| Osmium (Os) | $(NH_4)_2OsCl_6$ | Traceable to in-house standard, no NIST SRM available | ICP-OES |
| Palladium (Pd) | Pd metal | NIST SRM 3138 | ICP-OES |
| Platinum (Pt) | $H_2PtCl_6$ | NIST SRM 3140 | ICP-OES |
| Rhodium (Rh) | $RhCl_3$ | NIST SRM 3144 | ICP-OES |
| Ruthenium (Ru) | $RuCl_3$ x 3 $H_2O$ | Traceable to in-house standard, no NIST SRM available | ICP-OES |

## 4. Storing Behavior

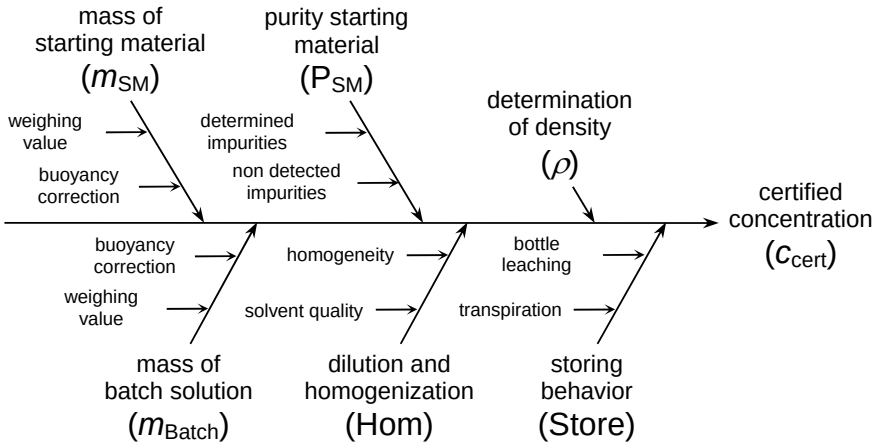
The storage behavior of standard solutions is an important factor regarding the certified value. An ideal container for a CRM solution is chemically inert, completely tight and does neither adsorb the analyte nor leach trace impurities into the solution. Therefore the most important storing effects were 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 the transpiration rate.


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

## 5. 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 [5]:




Typical contributions:

| | |
|---|---|
| $u(m_{SM})$ | < 0.01% |
| $u(m_{Batch})$ | < 0.01% |
| $u(P_{SM})$ | < 0.4% |
| $u$(Hom) | < 0.03% |
| $u$(Store) | < 0.09% |
| $u(\rho)$ | < 0.05% |

Combined uncertainty [6]:

| | |
|---|---|
| $u_c(c_{cert})$ | < 0.4% |

Expanded uncertainty:

| | |
|---|---|
| $\boldsymbol{U(c_{cert})}$ | **< 0.9%** |

CRM operations: S.Matt

S.Matt

Approving Officer: Claudia Geitner

C. Geitner, Ph.D.

**ISO Guide 34**
**SRMS 0001**

**ISO/IEC 17025**
**STS 0490**

**ISO 9001**
**005356 QM08**


[1] ISO Guide 31:2015, "Reference materials - Contents of certificates, labels and accompanying documentation"
[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] 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.
[6] Calculated by combination of the squared contribution values





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