

# Certificate

Produced in double accredited laboratory fulfilling  
**ISO/IEC 17025** and  
**ISO Guide 34**

This certificate is designed in accordance with ISO Guide 31<sup>[1]</sup>.

Object of certification: **Zinc standard for ICP**

Product No.: **18562**

Lot: **BCBN8385V**

Composition: Zinc metal (high purity quality) in 2% HNO<sub>3</sub> (prepared from HNO<sub>3</sub> *TraceSELECT*<sup>®</sup> and water *TraceSELECT*<sup>®</sup> Ultra, 18.2 MΩ·cm, 0.22 μm filtered)

Density at 20°C:  $\rho = 1011.4 \text{ kg m}^{-3}$        $u_c(\rho) = 0.5 \text{ kg m}^{-3}$

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

Storing and handling: This reference material shall be stored in the original closed bag between 5°C and 30°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 and the bottle should be stored at reduced temperature (e.g. refrigerator) to minimize transpiration rate. We highly recommend using this reference material no longer than 15 months after the aluminum bag was opened.

Expiry date: **SEP 2018** (unopened bottle in aluminized bag)

Certificate issue date 21 NOV 2014

Bottle opening date:  
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Certified value traceable to SI unit kg and uncertainty according to ISO Guide 35 <sup>[2]</sup> and Eurachem/CITAC Guide <sup>[3]</sup>		
Constituent	Certified value at 20°C and expanded uncertainty [ $U = k u_c$ ; $k = 2$ ]	
<b>Zinc</b>	<b>989 mg kg<sup>-1</sup> ± 2 mg kg<sup>-1</sup></b>	<b>1'000 mg L<sup>-1</sup> ± 2 mg L<sup>-1</sup></b>

## 1. CONCEPT OF CERTIFICATION AND TRACEABILITY STATEMENT

To guarantee top reliability of the values for this *TraceCERT*<sup>®</sup> certified reference material three independent procedures were followed. The values have to agree in the range of their uncertainties, but the impurity corrected value from the gravimetric preparation has been chosen as certified value<sup>[4]</sup>:

1. Gravimetric preparation using pure materials is a practical realization of concentration units, through conversion of masses and mole fraction to mass fraction<sup>[4]</sup>. 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 *TraceCERT*<sup>®</sup> reference materials is based on this approach and directly traceable to the SI unit kilogram.

Therefore comprehensively characterized materials of highest purity are used (see paragraph 2). All balances are certified by DKD and calibrated with OIML Class E2 (up to 12 kg) and F2 (up to 64 kg) weights. The bulk solution was homogenized by overhead tumbling in a PVDF container for at least 6 hours. A peristaltic pump with perfluorinated polymer tubings was used for bottling. Detailed information about the long-term stability of the bottled solution is given in paragraph 5 of this certificate.

2. The starting material is measured against a certified reference material (e.g. 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 *TraceCERT*<sup>®</sup> calibration solution is compared to a second reference which is independent from the first reference.

## 2. PURITY 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 at Sigma-Aldrich Laborchemikalien, Seelze (Germany).

Water *TraceSELECT*<sup>®</sup>Ultra (18.2 MΩ·cm; 0.22 μm filtered, all metallic traces at ng kg<sup>-1</sup>-level) and acid in *TraceSELECT*<sup>®</sup> quality was used for preparation.

## 3. TRACE IMPURITIES IN BOTTLED SOLUTION

Up to 75 trace impurities were determined with 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<sup>-1</sup> (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
<0.025	<0.005											<0.025	<0.005	<0.020	<0.020	n.a.	n.a.
K	Ca	Sc	Ti	V	Cr	Mn	Fe	Co	Ni	Cu	Zn	Ga	Ge	As	Se	Br	Kr
<0.050	0.030	<0.001	<0.001	<0.001	<0.001	<0.001	<0.010	<0.005	<0.001	0.010	m	<0.001	<0.001	<0.001	<0.005	n.a.	n.a.
Rb	Sr	Y	Zr	Nb	Mo	Tc	Ru	Rh	Pd	Ag	Cd	In	Sn	Sb	Te	I	Xe
<0.001	<0.005	<0.001	<0.001	<0.001	<0.001	n.a.	<0.001	<0.001	<0.001	0.002	<0.001	<0.001	<0.001	<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	<0.005	<0.001	<0.001	<0.001	<0.001	<0.001	<0.001	<0.001	<0.001	<0.001	<0.001	0.001	0.019	<0.001	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												

## 4. 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. 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 728 / complexometric titration

bottled solution: BAM 365 / complexometric titration

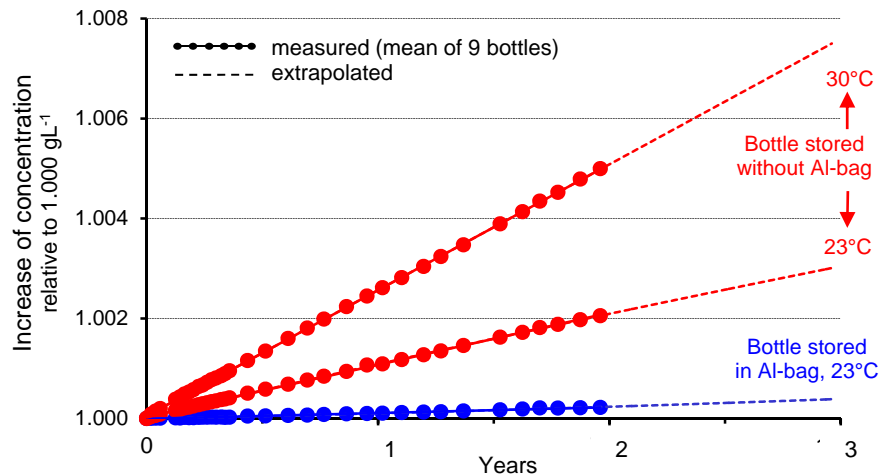
## 5. STORING BEHAVIOR

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 leach out of trace impurities from HDPE (high-density polyethylene) bottles was determined with HR-ICP-MS after leaching the bottles with 2% nitric acid. Maximum contamination levels were found in the  $\text{ng L}^{-1}$  level for 12 elements.

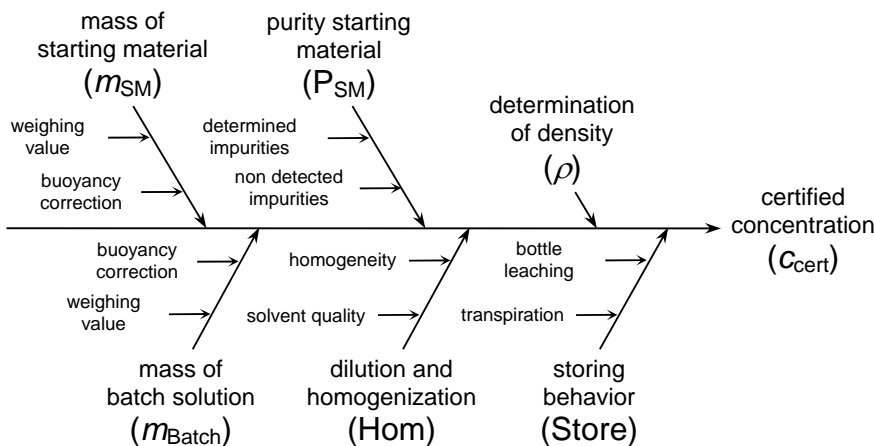
2. To avoid significant loss of mass through transpiration the bottle is delivered in aluminum coated bags. After the bottle has been removed from the bag, transpiration will occur at an accelerated rate (see figure). We highly recommend not to open the bag until the solution is needed. Once the bottle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

Transpiration through 100 mL HD-PE bottles



## 6. UNCERTAINTY EVALUATION

All uncertainties are calculated according to Eurachem/CITAC Guide <sup>[3]</sup> 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 <sup>[5]</sup>:



Typical contributions:

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

Combined uncertainty <sup>[6]</sup>:

$u_c(C_{cert})$	< 0.1 %
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Expanded uncertainty:

$U(C_{cert})$	< 0.2 %
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CRM operations:

*S. Matt*

S.Matt

Certification body:

*Klaus D. Schmidt*

K.-D. Schmidt, Ph.D.



ISO Guide 34



ISO/IEC 17025



ISO 9001

- [1] ISO Guide 31:2000, "Reference materials - Contents of certificates and labels"
- [2] ISO Guide 35:2006, "Reference materials - General and statistical principles for certification"
- [3] Eurachem/CITAC Guide, 3<sup>rd</sup> Ed. (2012), "Quantifying uncertainty in analytical measurement"
- [4] Eurachem/CITAC Guide, 1<sup>st</sup> 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