

# 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: **Rubidium standard for ICP**

Fluka product no.: 01444 (Lot 1388002, Filling Code 32608156)


Composition: Rubidium nitrate (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:  $\rho = 1010.2 \text{ kg m}^{-3}$        $u_c(\rho) = 0.5 \text{ kg m}^{-3}$

Intended use: Calibration of ICP-spectrometry, 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. After opening the bottle should be stored at reduced temperature to minimize transpiration rate. Before every use of the material the bottle must be shaken well and its temperature has to be 20°C. We highly recommend using this reference material no longer than 15 months after the aluminum bag was opened.

Expiry date: **03. June 2012** (unopened bottle in aluminized bag)

Bottle opening date: 

Certified value traceable to NIST SRM and uncertainty according to ISO Guide 35 <sup>[2]</sup> and Eurachem/CITAC Guide <sup>[3]</sup>		
Constituent	Certified value at 20°C <sup>[4]</sup>	Expanded uncertainty [ $U = k \cdot u_c$ ; $k = 2$ ]
<b>Rubidium</b>	<b>999 mg L<sup>-1</sup></b>	<b>4 mg L<sup>-1</sup></b>
	<b>989 mg kg<sup>-1</sup></b>	<b>4 mg kg<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 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<sup>[5]</sup>:

1. The content of the starting material is measured against an internationally recognized certified reference material (i.e. NIST, BAM or EMPA) followed by gravimetric preparation. Consequently the value calculated by this unbroken chain of comparisons is traceable to the reference to which the starting material is compared. 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. Whenever applicable the bottled TraceCERT<sup>®</sup> calibration solution is compared to a second reference (e.g. from NIST, BAM or EMPA) which is independent from the first reference.

## 2. CONTENT OF STARTING MATERIAL

The content of the starting material was established by ion chromatography measurements under ISO/IEC 17025:2005 (General requirements for the competence of testing and calibration laboratories).

The analysis result is directly traceable to the National Institute of Standards & Technology (NIST) Standard Reference Material 3145a, Rubidium Standard Solution. The uncertainty was calculated according to Eurachem/CITAC Guide<sup>[3]</sup> and is included in expanded uncertainty of the certified value.

## 3. TRACE IMPURITIES IN BOTTLED SOLUTION

Up to 75 trace impurities were determined with ICP-OES and ICP-MS. 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
<0.001	<0.001											<0.020	n.a.	n.a.	n.a.	n.a.	n.a.
Na	Mg											Al	Si	P	S	Cl	Ar
<0.050	<0.010											<0.050	<0.001	<0.001	0.020	n.a.	n.a.
K	Ca	Sc	Ti	V	Cr	Mn	Fe	Co	Ni	Cu	Zn	Ga	Ge	As	Se	Br	Kr
0.50	<0.010	<0.001	<0.015	<0.025	<0.025	0.027	<0.010	<0.005	<0.025	<0.010	0.023	<0.001	<0.050	<0.003	<0.003	n.a.	n.a.
Rb	Sr	Y	Zr	Nb	Mo	Tc	Ru	Rh	Pd	Ag	Cd	In	Sn	Sb	Te	I	Xe
m	<0.02	<0.015	<0.05	<0.001	<0.050	n.a.	<0.001	<0.001	<0.002	<0.010	<0.005	<0.003	<0.050	<0.001	<0.010	n.a.	n.a.
Cs	Ba	La	Hf	Ta	W	Re	Os	Ir	Pt	Au	Hg	Tl	Pb	Bi	Po	At	Rn
0.50	<0.010	<0.001	<0.001	<0.002	<0.003	<0.001	n.a.	<0.002	<0.001	<0.001	<0.002	<0.050	<0.050	<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	<0.002	<0.001	<0.002	<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), BAM (Germany) or EMPA – Material Science & Technology (Switzerland) 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. IC, ICP-OES or AAS.

Reference and applied technique used for traceability measurements of the starting material:

NIST SRM 3145 / Ion Chromatography

Reference and applied technique used for traceability measurements of the bottled solution:

NIST SRM 3145 / ICP-OES

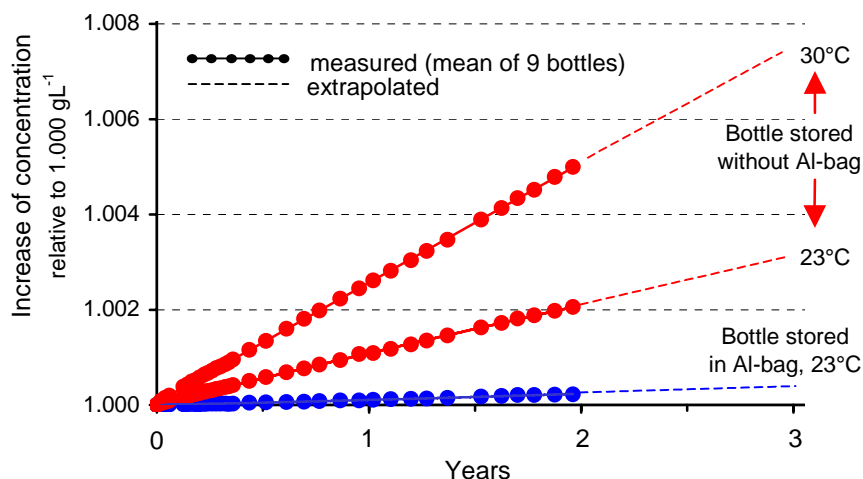
## 5. STORING BEHAVIOR

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 HD-PE 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.

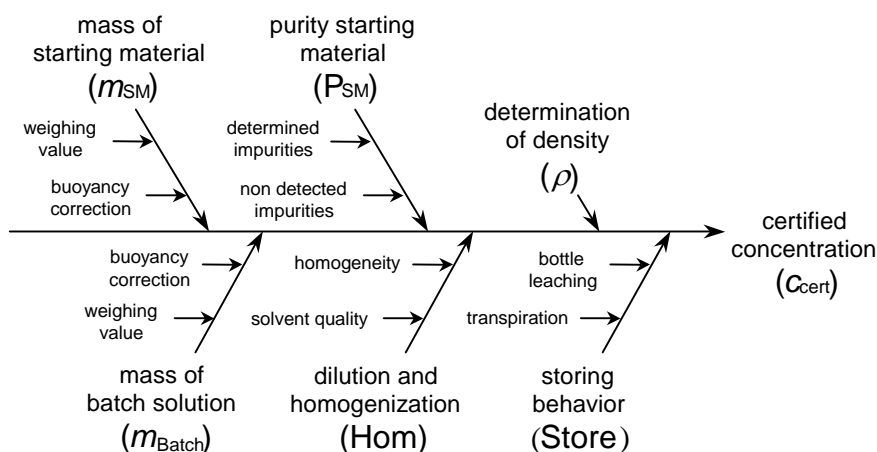
2. To avoid significant loss of mass through transpiration the bottle is delivered in aluminum coated bags. After the bottle has been removed from the bag, transpiration will occur at an accelerated rate (see figure). We highly recommend not to open the bag until the solution is needed. Once the bottle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

Transpiration through HD-PE bottles



## 6. 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 [6]:



Typical contributions:

$u(m_{SM})$	< 0.05%
$u(m_{Batch})$	< 0.05%
$u(P_{SM})$	< 0.15%
$u(Hom)$	< 0.03%
$u(Store)$	< 0.09%
$u(\rho)$	< 0.05%

Combined uncertainty [7]:

$$u_c(C_{cert}) < 0.2\%$$

Expanded uncertainty:

$$U(C_{cert}) < 0.4\%$$

Certification laboratory	Certification body	Date of release	Quality systems
 J. Wuethrich	 K.-D. Schmidt, Ph.D.	August 11 <sup>th</sup> 2008	

- [1] ISO Guide 31, 1-7, 2<sup>nd</sup> Ed. (2000), "Reference materials - Contents of certificates and labels"
- [2] ISO Guide 35, 1-64, 3<sup>rd</sup> Ed. (2006), "Reference materials - General and statistical principles for certification"
- [3] Eurachem/CITAC Guide, 1-120, 2<sup>nd</sup> Ed. (2000), "Quantifying uncertainty in analytical measurement"
- [4] The  $\text{mg kg}^{-1}$  value is the certified value whereas the  $\text{mg L}^{-1}$  value is calculated with the density
- [5] Eurachem/CITAC Guide, 1-37, 1<sup>st</sup> Ed. (2003) "Traceability in chemical measurement"
- [6] Reichmuth, A., Wunderli, S., Weber, M., Meyer, V. R., "The uncertainty of weighing data obtained with electronic analytical balances", *Microchimica Acta* (2004) 148: 133-141.
- [7] Calculated by combination of the squared contribution values