



**Český metrologický institut**

**Okružní 31, 638 00 Brno**

tel. +420 545 555 111, fax +420 545 222 728, www.cmi.cz

**Workplace:** Regional Branch Praha, Radiová 3, 102 00 Praha, Dept. Of gas mixture and reference materials certification, phone. +420 266 020 166, fax. +420 272 704 852

## **CERTIFICATE**

**1014-CM-E9001-10**

**CERTIFIED REFERENCE MATERIAL -**

**Aqueous Calibration Solution CZ 09001 ÷ CZ 09200**

**Date of issue:** 16.06.2010

page 1 (total 4)

**Valid until:** 16.06.2015

**Description and preparation:**

Aqueous calibration solutions of individual elements and ions with certified concentrations at the 1 g/l and 10 g/l levels and aqueous calibration solutions of combinations of elements and/or ions with certified concentrations at various levels relevant to particular analytical requirements.

**Purpose:**

The Certified Reference Materials are designed for calibration of instruments and providing traceability to SI unit. They can be used by analytical techniques analyzing aqueous solutions such as atomic spectrometry (AAS, AFS, ICP-AES, ICP-MS), molecular absorption spectrometry, ion chromatography, and some selected electroanalytical methods.

**Instruction of use:**

The attached Identification Sheet of a specific CRM of a particular batch is an integral part of the Certificate as a legal metrological document.

**Producer:**

ANALYTIKA<sup>®</sup> spol. s r.o, Ke Klíčovu 2a/816, 190 00 Praha 9  
Ing. Daniela Weissarová, E-mail:sales@analytika.net

**CMI responsible person:**

**Director of CMI OI Prague:**

Ing. Jan Beránek



Ing. Zdeněk Jirák  
zástupce ředitele OI Praha

Ing. Vladimír Peršl



**Preparation procedure:**

The mass of the starting primary substance (pure metal or compound) calculated with respect to an assay of the certified constituent and the target volume of the batch, is weighed on an analytical balance and transferred quantitatively to a tall 600 ÷ 800 ml beaker and dissolved completely in appropriate media (e.g. water, acid, acid mixture). The resulting solution is then transferred into a volumetric flask (with volume corresponding to the target volume of the batch - usually 5 or 10 l), containing reagents ultimately forming the designed matrix. After adding deionized water up to 90 ÷ 95% of the final volume the solution is tempered to  $20.0 \pm 0.1$  °C, filled up to the mark with similarly tempered water and thoroughly mixed. A sample is taken of the prepared solution and impurities determined. The solution, divided with a measuring cylinder into ultimate unit portions, is then filled into dry leached bottles which are closed with a polypropylene screw cap, provided with protective parafilm, labelled and stored. All data relevant to the particular CRM batch (e.g. source and declared assay of the primary substance, weighing, volume and temperature measurement, determination of impurities, codes, dates of the preparation steps and personnel involved, etc.) are duly recorded and filed in both written and electronic form at least until the certification expires. One unit of each batch is stored for a period of at least 5 years.

**Starting primary substances and other applied materials:**

High purity metals with a declared assay of minimum 99.99% are preferred, and compounds of a defined and constant stoichiometry and declared assay and/or impurities content are used when a suitable metal is not available.

The supplier of primary substances must comply with ISO Guide 34 [2], namely to employ a certified Quality Management System. Each supply of the primary substance must be accompanied with complete documentation, including an identification of sources and treatment, assay and impurity contents. As required by this Guide, the supplier's data are verified by the CRM producer and by other subcontracted accredited laboratories (e.g. trace metal impurities by spectroscopic methods such as ICP-AES, ICP-MS, AAS, the assay by primary gravimetric or volumetric methods). Acids (prepared by sub-boiling distillation by the CRM producer), other ultrapure chemicals (ammonium hydroxide, hydrogen peroxide, etc.) and deionised water with a specific conductivity below  $0.5 \mu\text{S}/\text{cm}$  are further applied in the CRM preparation. Their impurity contents are regularly monitored. They are generally negligible compared with the concentration of the certified constituent.

**Weighing, volume and temperature measurement:**

A Class 1 electromechanical analytical balance with a valid verification (type Sartorius 2004 MP) is used to weigh the primary substances. All volumetric vessels used (flasks, pipettes, burettes) are of Accuracy Class 1, calibrated by their producers. They are accompanied with certificates with an uncertainty of volume at  $20.0$  °C statement. Two mercury thermometers  $0 - 30$  °C (with a scale division by  $0.1$  °C), regularly calibrated by authorised institutions are used for the temperature measurement.

Český metrologický institut  
Oblastní inspektorát Praha  
Radiová 3  
102 00 Praha  
-2-



**Coding and packing:**

A four-digit code - CRM CZ 9001 through 9200 - identifies the certified constituent (constituents), whereas the alternative certified concentration level and matrix is differentiated in both the Identification Sheet and the label by an alphanumeric code (in parentheses). The certified concentrations 1.000g/l and 10.00 g/l are marked 1 and 10, respectively. The letter following denotes the matrix: C stands for a diluted HCl, N for a diluted HNO<sub>3</sub>, S a diluted H<sub>2</sub>SO<sub>4</sub>, F a diluted HF, H a H<sub>2</sub>O matrix, a combination of letters, e.g. C, N specifies a diluted HCl + HNO<sub>3</sub>, etc.

A basic packing unit is 500 ml (with options of 250 and 100 ml) in an amber HDPE bottle or an amber glass bottle whenever an interaction of the certified constituent with HDPE may be suspected (e.g. mercury, precious metals).

Prior to filling, the bottles and caps are leached by HCl (5% v/v) for a longer period, washed repeatedly with deionised water and thoroughly dried. After filling with CRM, the remaining volume above the liquid is purged by nitrogen, the bottle closed hermetically with a polypropylene screw cap and sealed with parafilm. The bottle is labelled with a resistant plastic label containing all data as required by CRM standards, i.e. CRM and batch codes, producer identification and contact data, expiry term and further data and warning symbols when required by specific regulations.

**Homogeneity and stability:**

The CRMs are considered homogeneous due to their physico-chemical character (diluted solutions) and thus homogeneity testing is not required.

An assumption of stability and the consequent expiration period is based on both over ten years experience with the preparation of similar RMs and a test of possible instability factors. These factors are of general (e.g. water evaporation by regular weighing of the filled bottle) and specific character (e.g. adsorption and desorption of the particular certified constituent on the walls of the bottle, precipitation, etc.) Along with these tests the content of the certified constituent in the bottle is compared (at least twice during the validity period) with that of a newly prepared batch and/or a corresponding CRM from other producers.

**Certification, certified values and their uncertainties:**

Certification of concentration values is based on the CRM preparation by ISO Guide 35 [3], i.e. on weighing and volume measurement. They are calculated from an assay of the certified constituent in the primary substance, declared by its supplier and duly verified by the CRM producer. The certified concentration (m/v) and its uncertainty are expressed in g/l at a temperature of 20±0.1 °C.

The uncertainty of a certified value is estimated in compliance with ISO and EURACHEM methodologies [4, 5]. It combines the calculated standard uncertainties of the individual CRM preparation steps with an expert estimate of the standard uncertainty of the assay of the certified constituent in the primary substance, usually a dominant contribution. The expanded combined uncertainty calculated using a coverage factor  $k = 2$  is expressed as a two-sided half-interval by one significant figure, with the certified value given by the same number of digits.

The particular certified values and uncertainties, mostly at a level of 0.2% (rel.), are specified in the Identification Sheets together with the expiry terms and other relevant information.

**Traceability and quality assurance:**

Traceability to SI unit (mol) is secured by using a primary substance with a verified content of the certified constituent and by the method of the CRM preparation - weighing and volume measurement.

The CRM producer has ISO 9001:2008 Quality Management System (QMS) in force. Moreover, the producer operates an own non-certified QMS covering relevant chapters from ISO Guide 34 (not included in ISO 9001).

**Ceský metrologický institut**  
Oblastní inspektorát Praha  
Radiová 3  
102 00 Praha  
-2-



**Instructions for use:** The selection of the concentration of a certified constituent and a matrix adequate to the particular analytical method, as well as the correct preparation of diluted calibration solutions and their proper application is entirely within the user's responsibility. CRMs should be stored in their original packing (in an upright position) at temperatures between 10 to 30 °C, away from any strong light source (e.g. sunshine, UV lamp). It is advised to record the first opening of the bottle, to close it immediately after every use and to discard the contents of the bottle when less than 10 percent of the original volume of the solution is left.

The expiry term is valid provided the CRM is properly stored and handled in compliance with these instructions. The certification ceases to be valid if the CRM is damaged, contaminated or modified. (Please note the difference between the validity period of the authorization of the producer and the expiry term of a particular batch of CRM given in its Identification Sheet).

The user is responsible for an update of his contact data with the CRM producer to facilitate immediate relay of information on possible expiry terms and other essential data changes.

Safety regulations for transportation, storage and use of CRM are specified in the Safety Data Sheet enclosed with each supply.

**References:**

1. J. R. Moody, R. R. Greenberg, K. W. Pratt, T. C. Rains (NIST U.S.A.): Analytical Chemistry, Vol. 60, No. 21, 1203A (1988). Recommended Inorganic Chemicals for Calibration.
2. ISO Guide 34: 2009, General Requirements for the Competence of Reference Material Producers.
3. ISO Guide 35: 2006, Certification of Reference Materials – General and Statistical Principles.
4. Document EURACHEM: 2000, Quantifying Uncertainty in Analytical Measurement.
5. ISO Guide to the Expression of Uncertainty in Measurement, ISBN 92-67-10188-9, Geneva 1993.
6. ISO/IEC 17025:2005, General Requirement for the Competence of Testing and Calibration Laboratories.
7. ISO Guide 30: 1992, Terms and Definitions Used in Connection with Reference Materials.
8. ISO Guide 31: 2000, Reference Materials – Contents of Certificates and Labels.
9. ISO Guide 33: 2000, Uses of Certified Reference Materials.
10. Document BCR/01/97: 1997, Guidelines for the Production and Certification of BCR Reference Materials.
11. Reference Materials for Environmental Analysis, ed. by R. E. Clement, L. H. Keith and K. W. Michael Siu, CRC Press Inc., 1997, Chapter 3: Preparation and Verification of ICP and AA Reference Standards.
12. ISO/IEC Guide 99:2007, International Vocabulary of Metrology-Basic and General Concepts and Associated Terms (VIM).
13. ISO 9001:2008, Quality Management Systems-Requirements.
14. Directive of CORM CMI, 017-MP-C001-06, Preparation and Certification of Reference Materials, Prague 2006.

End of the Certificate

Český metrologický institut  
Oblastní inspektorát Praha  
Radiová 3  
102 00 Praha  
-2-