DETAILED INFORMATION ABOUT THE PRODUCTION OF AQUEOUS CALIBRATION SOLUTIONS ASTASOL®

DESCRIPTION AND USE:

These reference materials are aqueous calibration solutions of individual elements and ions with certified/assigned concentration value in the range from 1 mg/l to 10 g/l or 1mg/kg to 10g/kg (m/m) and aqueous calibration solutions of combinations of elements and/or ions with certified/assigned concentrations at various levels relevant to particular analytical requirements. They can be used by analytical techniques analysing aqueous solutions such as atomic spectrometry (AAS, AFS, ICP-OES, ICP-MS), molecular absorption spectrometry, ion chromatography and some selected electroanalytical methods.

STARTING PRIMARY SUBSTANCES AND OTHER APPLIED MATERIALS:

Metals or compounds (in mass calculated from the content of the certified component and a target volume of the batch) are used as starting materials for the production of these RM. High purity metals with a declared assay of minimum 99.95 % 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 [1, 2]. The supplier of primary substances must comply with ISO 17034 [3], 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), other ultrapure chemicals (ammonium hydroxide, hydrogen peroxide, etc.) and deionised water with a resistivity 18 MΩ.cm are further applied in the (C)RM preparation. Their impurity contents are regularly monitored. They are generally negligible (~ 1 µg/l) as compared with the concentration of the certified/assigned constituent.

WEIGHING, VOLUME AND TEMPERATURE MEASUREMENT:

A class 1 electromechanical analytical balances with a valid verification is used to weigh the primary substances. A performance of this balance is regularly controlled by the Czech Metrology Institute. Mettler weights class F1 (5 g and 100 g) are used for an internal calibration control. Two calibrated mercury or alcoholic thermometers 0 – 50 °C (with a scale division by 0.1 °C), regularly calibrated by authorized institutions are used for the temperature measurement.

All volumetric vessels used (flasks, pipettes, burettes) are of Accuracy Class 1, calibrated by their producers and delivered with a relevant certificate. The calibration of all labware is regularly checked.

CODING AND PACKAGING:

(C)RMs are delivered in amber HDPE bottles or amber glass bottles whenever an interaction of the analyte 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. The bottle filled with (C)RM is hermetically closed with a polypropylene screw cap and tightened with parafilm immediately after filling and sealed into reclosable aluminium bag or PE

foil. The bottle is labelled with a resistant plastic label containing all data as required by (C)RM standards, i.e. (C)RM and batch codes, volume of the packed unit, type of the matrix, producer identification and contact data, further data and warning symbols when required by specific regulations [4, 5].

HOMOGENEITY AND STABILITY:

The (C)RM are considered homogeneous due to their physico-chemical character (diluted solutions) and thus a detailed homogeneity testing is not required. This fact is supported by a long-term experience of the producer and other recognized producers of similar materials. In compliance with a recommendation of ISO 17034, homogeneity testing is performed (as prescribed in ISO Guide 35) at randomly chosen solutions and evaluated by ANOVA.

An assumption of stability and the consequent expiration period is based on both over 20 year 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 analyte on the walls of the bottle, precipitation, etc.). Along with these tests the content of the certified/assigned constituent in the bottle is compared (at least twice during the validity period) with that of a newly prepared batch and/or a corresponding (C)RM form other producers. For these comparisons, both primary analytical methods (gravimetry, titrimetry) and some instrumental methods (ICP-OES, AAS) working in the regime of maximum precision (the application of internal standard, bracketing technique etc.) are used. For selected analytes, an accelerated isochronous stability test (in the temperature range of -20 °C to +40 °C) is realized. Based on these tests, transport conditions for a particular (C)RM are defined.

CERTIFICATION, CERTIFIED AND ASSIGNED VALUE AND THEIR UNCERTAINTIES:

The concentration value of CRM/RM are certified/assigned on the basis of gravimetric preparation (refering to ISO Guide 35), i.e. on weighing and volume measurement [6]. 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/assigned concentration (m/v) and its uncertainty are expressed in g/l (alternatively in mg/kg) at a temperature of 20 ± 0.1 °C.

The uncertainty of the certified/assigned values is estimated in compliance with ISO and EURACHEM methodologies [7, 8]. It combines the calculated standard uncertainties of the individual CRM/RM 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 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 Certificate together with the expiry terms and other relevant information.

The particular assigned values and uncertainties, mostly at a level of 0.5% (rel.), are specified in the Identification sheet together with the expiry terms and other relevant information.

The certified/assigned analyte concentration and its uncertainty is further experimentally verified by means of primary and instrumental analytical methods [9]. However, the results received are not used in the calculation of the certified/assigned value and serve only for the confirmation that this value is true.

TRACEABILITY AND QUALITY ASSURANCE:

Direct traceability to SI unit for the weight (1 kg) is secured by using a primary substance within comprehensively verified content of the analyte constituent and by gravimetric method of preparation. This traceability is for each produced batch of (C)RM further confirmed by analytical data acquired from the determination of certified analyte with validated primary and instrumental analytical methods. Only internationally accepted references (SRM NIST, TraceCERT etc.) are used.

The producer operates a certified quality management system ISO 9001 [10] and is accredited according to ISO 17034 [12].

Control analytical laboratory of the producer is accredited according to ISO/IEC 17025 [11].

STORING AND INSTRUCTIONS FOR USE:

This (C)RM must be stored in the original closed bottle (in an upright position) between 5 °C and 30 °C, away from any strong light sources (e.g. sunshine, UV lamp). The producer guarantees declared life time (shelf life) and expiration time (period of validity) provided (C)RM is properly stored and professionally handled. The temperature of the bottle with (C)RM must be 20 ± 0.5 °C before every use. After use, the bottle must be immediately tightly capped.It is recommended to tighten the screw cap with parafilm Do not pipette from the bottle. Do not return removed aliquots to bottle. The user should differentiate between the life time (shelf life) of the particular (C)RM (usually 5 years from the date of production) and expiration time (never more than 24 months from the first opening of the bottle). Both values are presented in the Certificate and Identification sheet. It is necessary to indicate on the Certificate/Identification sheet the date of the first opening of the bottle or aluminium bag and the expiration time, which depends on the date of the first opening. It is necessary to indicate the expiration time on the label of the bottle too.

It is not recommended to use the standard solution when the bottle contains less than 10 % of the solution. Therefore, in case of opaque bottle, it is important to indicate the amount of the solution used, e.g. on the label.

Transpiration through amber HDPE bottles (losses of the solution mass resulting in a gradual increase in the analyte mass fraction) was not observed. It is the producer's experience that the loss of vapour phase occurs solely around the screw cap of the bottle which is not airtight. Therefore, it is recommended to cover the screw cap and the neck of the bottle with parafilm layer after each opening and put the bottle into the reclosable aluminium bag, if it is delivered in.Safety regulations for transportation, storage, and use of this (C)RM are specified in the respective Safety Data Sheet.

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10. ČSN EN ISO 9001:XXXX: Quality Management Systems – Requirements.
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