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REFERENCE SHEET

CERTIFIED REFERENCE MATERIAL

IAEA-450

MASS FRACTIONS OF PLATINUM AND TRACE ELEMENTS IN ALGAE

Certified values for mass fractions

(based on dry mass)

Analyte	Certified value ¹ [mg kg ⁻¹]	Expanded uncertainty ² [mg kg ⁻¹]
As	0.102	0.007
Cd	0.158	0.010
Cr	1.48	0.12
Hg	0.104	0.012
Ni	0.82	0.10
Pb	0.98	0.07
Pt	0.074	0.004

⁽¹⁾ The certified values are given as total mass fractions of listed elements

⁽²⁾ The uncertainty is expressed as an expanded uncertainty using a coverage factor $k = 2$ estimated in accordance with the JCGM 100:2008 'Evaluation of measurement data – Guide to the expression of uncertainty in measurement' [1] and ISO Guide 35 'Reference materials – General and statistical principles for certification' [5]

Origin and preparation of the material

The IAEA-450 CRM is a unicellular microalga (*Scenedesmus obliquus*). Two batches of algae were grown in an outdoor bioreactor, one batch on a standard nutrient solution (natural contamination level), and the second batch on a nutrient solution containing elevated levels of As, Cd, Cr, Hg, Ni, Pb, and Pt. After harvesting, the bulk material was gamma-ray irradiated at the IAEA's laboratories to avoid a bacterial deterioration, and tested for homogeneity.

Thereafter, in order to adjust the Pt mass fraction to the level of environmental pollution in road dust, the two materials were mixed in a ratio of 1:100 (high Pt: low Pt). Using high purity methanol, slurries of the two algae materials were produced and merged under constant stirring in 200 g batches. After 72 hours drying at 60°C, the dry material was homogenized using a shaking device for 8 hours. Then the bulk material was combined/mixed into one batch and homogenized for 1 week in a 50 L drum homogenizer containing 20 ceramic balls of 2 cm diameter.

Bottling of IAEA-450 was carried out under normal laboratory conditions. Portions of 10 g were filled into plastic bottles, sealed with security polyethylene caps and labelled with the code IAEA-450. After bottling, the material was sterilized by gamma-ray irradiation with a total dose of 25 kGy using a ⁶⁰Co source according to EN ISO 13485:2003 in an irradiation facility.

Homogeneity of the material

Ten bottles of the IAEA-450 material were randomly selected from the 970 bottles produced for between and within bottle homogeneity studies. Three test portions of 0.2 g were taken from each bottle for the determination of Ni, Pb, As, Cd, Cr and Pt by ICP-MS. For Hg analysis, a Direct Mercury Analyser (DMA) using atomic absorption spectrophotometry at 254 nm was used.

Three test portions of 0.2 g were taken from each bottle for the determination of As, Cd, and Cr by Octopole Reaction Cell-ICP-MS and for Ni and Pb by ICP-MS. Low level Pt was determined using reverse ID-ICP-MS.

The analysis of homogeneity study was performed under repeatability conditions to minimize variations. The homogeneity test results provided experimental evidence that satisfactory levels of between and within bottle homogeneity were attained and the uncertainties due to between and within bottle heterogeneity were less than 5%. Thus the material was considered sufficiently homogeneous for the tested elements [2].

Dry mass determination

The average moisture content of the material was determined by drying several test portions of 0.5 g in an oven at 80°C for 12 hours, and was found to be approximately 4.7 (±0.2)%. Since the moisture content can vary with ambient humidity and temperature, it is recommended to check it prior to analysis and to report all results on a dry mass basis.

Characterization study

This material was thoroughly characterized by group of national metrological laboratories. The certified mass fractions are based on measurement results obtained from national metrological laboratories using validated methods with known accuracy and sufficient traceability. ICP-MS technique was mainly used by all laboratories and applied isotope dilution techniques for Ni, Pt, Cd, Pb and Cr. NAA technique was used by few laboratories for As, Cr measurements and one laboratory used cold vapor atomic absorption spectrometry for Hg analysis.

Assignment of values – Certification procedure

The certified values were derived as an Algorithm A as described in Annex C of ISO 13528 [4]. Mean and median were also calculated for comparison purposes. These values were compared with the calculated respective Algorithm A values and no significant difference was observed which ensures reliable estimation of assigned value.

The uncertainties associated with the reference values were calculated according to the JCGM 100:2008 Evaluation of measurement data – Guide to the expression of uncertainty in measurement [1] and ISO Guide 35 [5]. The combined uncertainty of the certified values consists of uncertainty components associated with characterization (u_{char}), between bottle heterogeneity (u_{bb}) and stability (u_{stab}). These different contributions were combined to estimate the combined uncertainty.

The IAEA Reference Materials Certification Committee decided to accept the assigned values as presented in the table above.

Statement on metrological traceability and uncertainty of assigned values

The quantity values assigned to the certified reference material are mass fractions of specified elements, expressed in the derived SI unit mg kg^{-1} . Values were derived from individual results reported by the metrological institutes [3]. Evidence on metrological traceability to the SI Units was provided for all results taken into account for the calculation of the assigned values.

Intended use

This certified reference material is suitable for quality assurance and quality control purposes in determination of mass fractions of listed elements, when samples of similar matrix composition are analyzed. The IAEA-450 CRM is also suitable for method development and all aspects of analytical method validation, including potential bias evaluation, and for training purposes.

Instructions for use

The certified reference material is supplied in 10 g units. The material homogeneity is guaranteed if a minimum test portion of 0.2 g is used.

To overcome segregation effects due to storage or transportation, the material should be mixed before opening the bottle. All necessary precautions should be taken when opening the bottle to prevent any spread of the fine powder in the laboratory.

Handling and storage

The original unopened bottle should be stored securely at ambient temperature in a dark and dry place. Analysts are reminded to take appropriate precaution in order to avoid contamination of the material during handling.

Issue and expiry date

The issue date of this Certified Reference Material is **March 2012**; the certificate was revised in **September 2015**. Based on experience with similar materials, the reference values for studied elements are valid until **September 2025**, provided the original bottle is handled and stored in accordance with the instructions given in this reference sheet (see “Handling and Storage”).

Legal disclaimer

The IAEA makes no warranties, expressed or implied, with respect to the data contained in this reference sheet and shall not be liable for any damage that may result from the use of such data.

Compliance with ISO Guide 31:2000

The content of this IAEA Reference Sheet is in compliance with the ISO Guide 31:2000: Reference materials – Content of certificates and labels [6].

It is suggested to cite this reference sheet according to the following example, as appropriate to the citation format used: INTERNATIONAL ATOMIC ENERGY AGENCY, Reference Sheet for CRM IAEA-450, 'Mass fractions of platinum and trace elements in algae', IAEA, Vienna, 4 pp. (The latest version published applies; see "Note" below).

Note

Certified values as stated in this reference sheet may be updated if more information becomes available. Users of this material should ensure that the reference sheet in their possession is current. The current version can be found in the IAEA's Reference Materials online catalogue:

<http://nucleus.iaea.org/rpst/ReferenceProducts/ReferenceMaterials>

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REFERENCES

- [1] JOINT COMMITTEE FOR GUIDES IN METROLOGY (JCGM), Evaluation of measurement data - Guide to the expression of uncertainty in measurement, JCGM 100:2008 (GUM with minor corrections), (2008). http://www.bipm.org/utls/common/documents/jcgm/JCGM_100_2008_E.pdf
- [2] SATHRUGNAN, K., Certification of mass fractions of platinum and trace elements in CRM IAEA-450 (algae) sample, Internal Report NAEL/RM/002 (September 2015).
- [3] SHAKHASHIRO, A., TOERVENYI, A., GAUDINO, S., ROSAMILIA, S., BELL, M., TURK, G.C., CCQM key comparison CCQM-K75: K75 Determination of toxic metals in algae. Draft, Metrologia **48** Technical Supplement, **08002** (2011).
- [4] INTERNATIONAL ORGANIZATION FOR STANDARDIZATION, Statistical methods for use in proficiency testing by interlaboratory comparisons, ISO 13528:2005, ISO, Geneva (2005).
- [5] INTERNATIONAL ORGANIZATION FOR STANDARDIZATION, Reference materials: general and statistical principles for certification, ISO Guide 35:2006, ISO, Geneva (2006).
- [6] INTERNATIONAL ORGANIZATION FOR STANDARDIZATION, Reference materials — Content of certificates and labels, ISO Guide 31: 2000, ISO, Geneva (2000).



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Revision information:

- Original reference sheet issue date: 2012-02-24
- Rev.01 (2015-09-22): Table 1, Correction of values for expanded uncertainty (corrected for all analytes)); added more information on characterization and assignment of values; references were updated (Certification report NAEL/RM/002)