IAEA
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REFERENCE SHEET

REFERENCE MATERIAL

IAEA-413

MAJOR, MINOR AND TRACE ELEMENTS IN ALGAE

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Reference Values
(Based on dry mass)

Element	Reference Value Mass fraction [mg kg^{-1}]	Standard uncertainty* [mg kg^{-1}]	n**	Analytical Methods
As	127	6.6	10	INAA (3), HG-AAS, ICP-OES (2), AAS, XRF, ICP-MS (2)
Ca	3143	112	7	INAA, AAS, ICP-OES(3), XRF, ICP-MS
Cd	204	8.5	10	INAA(3), ICP-OES(3), AAS, XRF, ICP-MS(2)
Co	4.24	0.25	8	INAA(3), ICP-OES, ASV, AAS, ICP-MS(2)
Cr	377	14	10	INAA(3), ICP-OES, AAS, XRF, ICP-MS(2)
Fe	1370	39	9	INAA(3), ICP-OES(3), AAS, ICP-MS(2)
K	10740	270	6	INAA(2), ICP-OES(2), AAS, XRF
Mg	4058	117	5	ICP-OES(3), AAS, ICP-MS
Mn	158	3.4	7	INAA, ICP-OES(3), ICP-MS (2), AAS
Na	375	20	5	INAA(2), ICP-OES(2), AAS
Ni	113	4.9	9	ICP-OES(2), AAS, ICP-MS(2), ASV, INAA(3)
Pb	242	7	7	ICP-OES(3), AAS, ICP-MS(2), XRF
Zn	169	3.3	8	INAA(2), ICP-OES(2), AAS, XRF, ICP-MS (2)

* Uncertainty is expressed as a Mixture model median based standard deviation S(MM-median) at 95 % confidence level [1].
**number of accepted laboratory means which were used to calculate the reference values.

Information Values

(Based on dry mass)

Element	Information Value Mass fraction mg kg^{-1}	Standard uncertainty* mg kg^{-1}	n**	Analytical Methods
Cu	11.1	0.5	5	ICP-OES(3), AAS, ICP-MS
Hg	53.2	4.0	5	INAA(2), AAS CV(2), ICP-MS

*Uncertainty is expressed as a Mixture model median based standard deviation S(MM-median) at 95 % confidence level [1].
**number of accepted laboratory means which were used to calculate the reference values.

The reference and information values of all elements were established on the basis of a robust approach proposed by David L. Duewer [1] and the Mixture Model Median (MM-median) of the analytical results reported by the expert laboratories. The MM-median is a direct analogue of the median. It is the location which divides the Mixture Model Probability Density Function (MM-PDF) into two sections of equal area.

To estimate the standard uncertainty associated with the property value the MM-median based Standard Deviation S(MM-median) was calculated from the span of the central 50% of the MM-PDF density. The uncertainty related to long- and short-term stability was not considered.

The property values assigned to the algae IAEA-413 reference material are element mass fractions, expressed in the derived SI unit mg/kg. The utmost care was taken regarding the metrological traceability of the property values assigned to this reference material already at the planning phase and during the entire characterization process. Laboratories participating in the characterization campaign have been requested to carefully choose the calibrants and to provide the IAEA with all related information, including certificates. However, the selection of measurement methods and measurement procedures, as well as respective calibrants, was based on a decision of the participating laboratory. A consequence of the use of different calibrants, is the fact that the metrological chain(s) for each of the assigned quantity values respectively (combined from number of results), cannot not easily be described. Therefore, the assigned property values – the element mass fractions – although expressed in the derived SI unit, are not intended for calibration purposes, and the reference material as such is not to be used as a calibrant.

Further details on the characterization and calculation of property and uncertainty values can be found in the report IAEA/AQ/14, 'Reference material IAEA-413: Major, minor and trace elements in algae' [2].

Intended Use

This reference material is intended to be used for quality assurance purposes, basically as a quality control material for the measurement of the elemental composition of biological materials especially of biomonitors, for the assessment of a laboratory's analytical work and for the validation of analytical methods.
The estimated standard uncertainty is relatively high due to the consideration of the between laboratories dispersion in the calculation of the S(MM-Median). The user should establish his own reproducibility standard deviation to use it as a control limit for precision.

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.

Origin and preparation of the material

The IAEA-413 Algae reference material was prepared by the IAEA Terrestrial Environment Laboratory in Austria in co-operation with the Institute of Microbiology, Academy of Sciences of the Czech Republic in Trebon.
The IAEA-413 algae material (type: Chlorella Boehm) was produced under standard outdoor culture conditions. The bulk algae material and the sealed bottles were treated with gamma ray irradiation with a total dose of 25 kGy using a Co-60 source to improve long-term stability of the material by reducing microbial action.

Homogeneity

Sample bottles covering the whole bottling range were randomly selected for homogeneity tests and the characterization. During the homogeneity tests emphasis was placed on performing measurements under good repeatability conditions.
The following Table shows the relative repeatability standard deviation (RSD%) of INAA measurements obtained for 15 bottles. Sample mass used as test portion was 10 mg. Details can be found in the certification report [1].

Element	As	Br	Cd	Co	Cr	Fe	Hg	Na	Zn
RSD %:	1.17	1.18	1.24	2.49	0.94	2.25	1.55	1.13	1.74

No systematic attempt was made to evaluate the minimum representative sample size for IAEA-413. However, subsequent analysis for some elements by INAA indicates that even lower sample masses as 100 to 200 mg give comparable results.

Dry mass determination

All reference and information values are expressed on a dry mass basis. Therefore the analytical results need to be corrected for the moisture content of the sample at the time of analysis.

It is recommended to dry a separate test portion of at least 500 mg for 4 hours at 80°C. If smaller test portion masses are used, or the drying procedure is modified, the uncertainty on the dry mass factor is increased and should be taken into account for the total uncertainty calculation.

Instructions for use

The recommended minimum test portion is 200 mg. Before each sub-sampling, the bottle should be thoroughly shaken for to re-homogenize the sample again. Analysts are reminded to take appropriate precautions to avoid contamination of the sample and the remaining material in the bottle.

Storage

The original unopened bottle should be stored securely at ambient temperature in a dark and dry place. It is recommended to store the material after opening of the bottle in a refrigerator (at 4 to 8°C) or in a dessicator. Exposure to sunlight should be avoided. Storage at room temperature and even temperatures up to 40°C did not show degradation of the originally sealed algae material.

Expiration of the reference sheet

Taking into account previous experience with irradiated biological reference materials and the results of the stability tests performed and expert judgment, provided the original bottle is handled and stored in accordance with the instructions given in this reference sheet (see “Storage”). This certification is nullified if the bottle is damaged. Reference values as stated in this reference sheet

may be updated if more information becomes available. Users of this reference material should ensure that the reference sheet in their possession is current. This can be accomplished by accessing the appropriate web page at: http://www.iaea.org/.

The IAEA is monitoring the long term stability of the material and customers will be informed in case of any observed change.

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.

Legal disclaimer

Although great care has been taken to maintain the accuracy of information contained in this reference sheet, the IAEA assumes no responsibility for consequences which may arise from its use.

References

[1] DUEWER, D, L, A Robust Approach for the Determination of CCQM Key Comparison Values and Uncertainties, Paper presented at the 10th meeting of the CIPM Consultative Committee for Amount of Substance - Metrology in Chemistry, Sevres, France, (2004). www.bipm.info/cc/ccqm/allowed/10/ccqm04-15.pdf.

[2] ZEILLER, E., SHAKHASHIRO, A., TORVENYI A., FAJGELJ, A.: IAEA/AQ/14, Reference material IAEA-413: Major, minor and trace elements in algae, IAEA, Vienna, Austria, 2010.

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