International Atomic Energy Agency
Analytical Quality Control Services
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REFERENCE SHEET

REFERENCE MATERIAL

IAEA-392

TRACE, MINOR AND MAJOR ELEMENTS IN ALGAE

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Recommended Values
(Based on dry mass)

Element	Recommended Value Mass fraction mg/kg	Expanded uncertainty U k=2	n*	Analytical Methods
Ca	2680	67.4	6	INAA , AAS, ICP-OES
Cu	23.2	1.74	6	INAA, ICP-OES, AAS, ICP-MS
Fe	497	13.6	8	INAA, ICP-OES, AAS, ICP-MS
Mg	2376	78.8	4	ICP-OES, AAS, ICP-MS
Mn	67.5	1.54	5	INAA, ICP-OES, ICP-MS
Na	680	23.0	5	INAA, ICP-OES, AAS
Ni	0.571	0.028	4	ICP-OES, AAS, ICP-MS, DPCSV
Pb	0.574	0.019	4	IDMS, AAS, ICP-MS, DPCSV
Zn	128	2.0	8	INAA, ICP-OES, AAS, DPCSV

Information Values
(Based on dry mass)

Element	Recommended Value Mass fraction mg/kg	Expanded uncertainty U k=2	n*	Analytical Methods
As	0.175	0.016	4	INAA, HG-AAS
Cd	0.0173	0.0014	3	DPASV, IDMS, ET AAS
Co	3.33	0.122	7	INAA, ICP-OES, DPCSV, ICP-MS
Cr	4.57	0.180	7	INAA, ICP-OES, ET AAS, ICP-MS
K	8383	252.4	5	INAA, ICP-OES, AAS

**n = number of accepted laboratory means which were used to calculate the recommended or information values*

The recommended and information values are the unweighted means of n laboratory means. The uncertainties were calculated following recommendations given in ISO Guide 35 and include the uncertainty components derived from the sample heterogeneity at masses between 100 to 200 mg and the between laboratory variability. For the expanded uncertainty a coverage factor of two was applied (95% confidence interval). The uncertainty related to long- and short-term stability was not considered.

The property values assigned to the algae 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 characterisation and calculation of property and uncertainty values can be found in the report IAEA/AL/149 'Report on the characterisation of the naturally grown algae reference material IAEA 392' [1].

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.

Origin and preparation of the material

The IAEA-392 Algae material was prepared by the Analytical Quality Control Services of the International Atomic Energy Agency in co-operation with the Institute of Microbiology, Academy of Sciences of the Czech Republic in Trebon, during 1995.

The IAEA-392 algae material (type: Scenedesmus obliquus 208) was produced under standard outdoor culture conditions [2, 3]. Mineral nutrients were prepared from p.a. chemicals and added as required to maintain the optimum balanced composition of the nutrient medium in the growing culture. After harvesting, the algae were stored in a coupled tank and continuously spray dried at moderate venting and temperature avoiding a damage of the cell structure.

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

The natural cell particle size of the algae reference material IAEA 392 is about 8 µm. This small particle size by itself is a good basis for high sample homogeneity. During several homogeneity studies the uncertainty due to sample heterogeneity for sample masses between 100 to 200 mg was evaluated for all elements. The uncertainty was found not to exceed 2% for all elements except for Cu (2.8%). These values are already conservative since the uncertainty due to method repeatability was still partly included. Details can be found in the characterisation report [1].

No systematic attempt was made to evaluate the minimum representative sample size for IAEA-392. 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 recommended and information values are expressed on a dry weight 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 sample of at least 500 mg for 4 hours at 80°C. If smaller sample intake 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. An indication on the magnitude of the dry mass uncertainties and their influence on the final results can be found in a related publication [4].

Instructions for use

The recommended minimum sample intake is 200 mg. Before each subsampling, the bottle should be thoroughly shaken for 5 minutes to re-homogenise the sample again. Analysts are reminded to take appropriate precautions to avoid contamination of the sample and the remaining material in the bottle. 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, the expiration date of the reference sheet has been set to December 2009. The reference values are valid within the stated uncertainties, provided that the Reference Material is handled in accordance with the instructions for use. The IAEA will further monitor the material and will immediately inform the clients of any factors, which are found to affect the contents of the reference sheet and its expiration date.

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.

References

[1] Zeiller E., A. Shakhashiro: IAEA/AL/149 'Report on the Characterization of the Naturally Grown Algae Reference Material IAEA 392, IAEA, Vienna, Austria, 2005

[2] Doucha J. et al.: Report on preparation of microalgae biomass with different content of heavy metals, Trebon, Czech Republic, 1996.

[3] Doucha J., Lívansky K.: Novel outdoor thin layer high density microalgae culture system: Productivity and operational parameters, Arch. Hydrobiol. 106/Algolog. Stud. 76, 129-147 (1995).

[4] Zeiller E., Benetka E., Koller M., Schorn R.: Dry mass determination - which role does it play for the combined measurement uncertainty? A case study using IAEA algae reference materials (presented at the IUPAC Symposium on 'Trace elements in food' Oct 2004, paper submitted for publication)

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