

**International Atomic Energy Agency
Department of Nuclear Sciences and Applications
IAEA Environment Laboratories**

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REFERENCE SHEET

CERTIFIED REFERENCE MATERIAL

IAEA-461

**MASS FRACTIONS OF TRACE ELEMENTS AND METHYL MERCURY IN
MARINE BIOTA (Clam, *Gafrarium tumidum*) SAMPLE**

Certified mass fraction values
(based on dry mass)

Analyte	Unit	Certified value^(a)	Expanded uncertainty^(b)
As	mg kg ⁻¹	30.2	1.5
Ca	mg kg ⁻¹	9.27 x10 ³	0.90 x10 ³
Cd	mg kg ⁻¹	0.569	0.040
CH ₃ Hg	mg kg ⁻¹ as Hg	62.3 x10 ⁻³	6.9 x10 ⁻³
Co	mg kg ⁻¹	4.78	0.60

(a) Information on calculation of certified values

Certified values are calculated from the accepted data sets, each set being obtained by a different laboratory and/or a different method of determination following ISO Guide 35 'Reference materials – General and statistical principles for certification' [1].

(b) Information on method used for uncertainty estimation

The uncertainty is expressed as a combined uncertainty with a coverage factor $k = 2$, corresponding to a level of probability 95%, estimated in accordance with the JCGM 100:2008 'Evaluation of measurement data – Guide to the expression of uncertainty in measurement' [2] and ISO Guide 35 [1].

Certified mass fraction values (contd.)
(based on dry mass)

Analyte	Unit	Certified value ^(a)	Expanded uncertainty ^(b)
Cr	mg kg ⁻¹	27.0	2.4
Cu	mg kg ⁻¹	26.1	2.0
Fe	mg kg ⁻¹	2.60 x10 ³	0.22 x10 ³
Hg	mg kg ⁻¹	0.390	0.040
Mn	mg kg ⁻¹	333	20
Ni	mg kg ⁻¹	106	8
Pb	mg kg ⁻¹	25.8	1.7
Se	mg kg ⁻¹	2.78	0.31
V	mg kg ⁻¹	6.86	0.67
Zn	mg kg ⁻¹	156	11

(a) Information on calculation of certified values

Certified values are calculated from the accepted data sets, each set being obtained by a different laboratory and/or a different method of determination following ISO Guide 35 'Reference materials – General and statistical principles for certification' [1].

(b) Information on method used for uncertainty estimation

The uncertainty is expressed as a combined uncertainty with a coverage factor $k = 2$, corresponding to a level of probability 95%, estimated in accordance with the JCGM 100:2008 'Evaluation of measurement data – Guide to the expression of uncertainty in measurement' [2] and ISO Guide 35 [1].

Information mass fraction values
(based on dry mass)

Analyte	Unit	Information value ^(a)	Expanded uncertainty ^(b)
Al	mg kg ⁻¹	870	160
Mg	mg kg ⁻¹	6.48 x10 ³	0.69 x10 ³
Sr	mg kg ⁻¹	112	7

(a) Information on calculation of information values

Information values are calculated from the accepted data sets, each set being obtained by a different laboratory and/or a different method of determination following ISO Guide 35 'Reference materials – General and statistical principles for certification' [1].

(b) Information on method used for uncertainty estimation

The uncertainty is expressed as a combined uncertainty with a coverage factor $k = 2$, corresponding to a level of probability 95%, estimated in accordance with the JCGM 100:2008 'Evaluation of measurement data – Guide to the expression of uncertainty in measurement' [2] and ISO Guide 35 [1].

Origin and preparation of the material

Sixty kg of clams (*Gafrarium tumidum*) were collected in Noumea, New Caledonia. Clams were opened, the soft-shell part was frozen and then freeze dried. The dry material was first ground and sieved at 250 µm. A portion of about 5 kg was micronized to achieve a final powder size <50 µm. The homogenization was performed by mixing the material in a stainless steel rotating homogenizer Moritz ERM-BB124 (Moritz, Chatou, France) for 14 days at a temperature of 20 (±2)°C and relative humidity of 50%. After checking for the homogeneity of sample material, aliquots of about 8 g were packed into pre-cleaned brown borosilicate glass bottles with polyethylene caps and then sealed in plastic bags. The sample material was labelled as IAEA-461.

Homogeneity of the material

The between-bottle homogeneity was tested by the determination of the mass fractions of selected certified analytes (Cr, Cu, Hg, CH₃Hg, Mn, Ni, Pb and Zn). In total, 11 bottles were selected using random stratified sampling of the whole batch. Three subsamples from each bottle were analysed for their total element mass fractions. For all elements except Hg and CH₃Hg, subsamples of 0.2 g were mineralized with 5 ml HNO₃ in a microwave oven. The measurements were performed by flame and graphite furnace atomic absorption spectrometry. The determination of total Hg was done in solid subsamples (50mg) with solid mercury analyser, and methyl mercury was determined by gas chromatography coupled with atomic fluorescence spectrometer (GC-AFS) after alkaline digestion and room temperature derivatization.

The measurements of homogeneity study was performed under repeatability conditions and in a randomized way in order to separate a potential analytical drift from a trend in the filling sequence and minimize variations. The homogeneity test results provided experimental evidence that satisfactory levels of between and within-bottles homogeneity have been attained and the uncertainty due to the between and within-bottles heterogeneity were within acceptable limits.

Characterization study

The assigned values were established on the basis of results reported to the IAEA-MESL by 18 laboratories (25 data sets).

Laboratories were requested to report 3 independent results of Al, As, Ca, Cd, Co, Cr, Cu, Fe, Hg, Mg, Mn, Ni, Pb, Se, Sr, V, Zn and CH₃Hg mass fraction with one of the following analytical techniques: Atomic-Absorption Spectrometry (Flame and Graphite Furnace), Inductively-Coupled Plasma Mass Spectrometry, Isotope Dilution Inductively-Coupled Plasma Mass Spectrometry, Neutron-Activation Analysis, Atomic Fluorescence Spectrometry and X-ray Fluorescence.

Assignment of values – Certification procedure

The assigned property values were established on the basis of results reported by participating laboratories to the IAEA-MESL. The robust mean concentrations for the sets of individual data were chosen as the best estimate of the property values [3]. A certified value was assigned when at least 5 independent results obtained by a minimum of 3 independent analytical methods and the relative expanded uncertainty was less than 15%. For Al, Mg and Sr criteria were not fulfilled; robust mean and uncertainties for those elements are given only as information values.

The details concerning all reported results as well as the criteria for certification can be found in [3] The report may be downloaded free of charge from:

https://www-pub.iaea.org/MTCD/Publications/PDF/IAEA-AQ-44_web.pdf

Based on the evidence on calibrators used, quality control procedures applied by the participating laboratories and their generally high quality performance in previous IAEA interlaboratory comparisons, the Certification Committee decided to accept these assigned values as certified as presented in the Table above.

Statement on metrological traceability, commutability and uncertainty of assigned values

The property values assigned to the IAEA-461 reference material are calculated as mass fractions of specified trace elements, expressed in the derived SI unit mg kg⁻¹. Certified values are SI traceable and evidence on their metrological traceability to the SI units may be found in reference [3]. Expanded uncertainties with a coverage factor of $k = 2$, corresponding to a level of confidence of about 95 %, were calculated according to the JCGM 100:2008 Evaluation of measurement data – Guide to the expression of uncertainty in measurement [1]. The measurement uncertainty associated with the certified value is expressed as mg kg⁻¹.

The agreement between results obtained with different analytical methods selected for the IAEA-461 characterization study confirms the absence of any significant method bias and demonstrates commutability of the material for all certified trace elements. More information on commutability of certified values may be found in reference [3].

Intended use

This Certified Reference Material is intended to be used for quality assurance and quality control purposes. The IAEA-461 Certified Reference Material is also suitable for method development and validation of analytical procedures, including potential bias evaluation, and for training purposes.

Instructions for use

The IAEA-461 Certified Reference Material is supplied in 8 g units. The material homogeneity is guaranteed if a minimum test portion of 0.2 g is used for As, Ca, Cd, Co, Cr, Cu, Fe, Hg, Mn, Ni, Pb, Se, V, Zn and CH₃Hg and 0.05g for total Hg.

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.

Dry mass determination

The average moisture content of the material was determined by drying several test portions of 1 g in an oven at 85°C until constant mass and was found to be $(6.0 \pm 0.5) \% (k=2)$. Since the moisture content may 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.

Handling and storage

The original unopened bottle should be stored securely at ambient temperature in a dark and dry place. It is recommended to avoid direct exposure to sunlight or to a source of heat.

Issue and validity date

The issue date of this Certified Reference Material is **20 May 2017**. The material was re-assessed in **September 2020** by long term stability test, confirming that there are no changes in the assigned property values. The expiration date as therefore been extended to **October 2025**. The IAEA is monitoring the long term stability of the material and customers will be informed in case of any observed change.

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 31:2015

The content of this IAEA Reference Sheet is in compliance with the ISO Guide 31:2015, Reference materials – Contents of certificates, labels and accompanying documentation [4].

Citation of this reference sheet

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-461, Mass fractions of Trace Elements and Methyl Mercury in Marine Biota (Clam, *Gafrarium Tumidum*) Sample, IAEA, Vienna, 6 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 may be found in the IAEA’s Reference Materials online catalogue:

<https://nucleus.iaea.org/sites/ReferenceMaterials/Pages/IAEA-461.aspx>

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http://www.bipm.org/utis/common/documents/jcgm/JCGM_100_2008_E.pdf
- [3] INTERNATIONAL ATOMIC ENERGY AGENCY, Certification of Trace Element Mass Fractions and Methyl Mercury in IAEA-461 Marine Biota (Clam, *Gafrarium Tumidum*) Sample, IAEA Analytical Quality in Nuclear Applications Series No. 44 (IAEA/AQ/44), IAEA, Vienna 2016
- [4] INTERNATIONAL ORGANIZATION FOR STANDARDIZATION, Reference materials – Contents of certificates, labels and accompanying documentation, ISO Guide 31: 2015, ISO, Geneva (2015).

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Revision Information:

- Original reference sheet issue date: 2017-05-20
- Rev.00.01 (2020-04-27): update of web link to the online catalogue in section 'Note'
- Rev 01 (2020-10-01): change of expiry date confirmed by long term stability test