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REFERENCE SHEET

CERTIFIED REFERENCE MATERIAL

IAEA-452

MASS FRACTIONS OF TRACE ELEMENTS AND METHYL MERCURY IN BIOTA SCALLOP (*Pecten maximus*)

Certified values for mass fractions

(Based on dry mass)

| Analyte | Certified value ¹ [mg kg ⁻¹] | Expanded uncertainty ² [mg kg ⁻¹] |
|---------|--|---|
| As | 17.9 | 1.1 |
| Cd | 30.5 | 1.6 |
| Cr | 5.2 | 0.7 |
| Cu | 11.0 | 0.8 |
| Fe | 1068 | 47.0 |
| Hg | 0.16 | 0.02 |
| Mn | 279 | 14.0 |
| Pb | 2.4 | 0.3 |
| Zn | 167 | 10.0 |
| MeHg | 0.022 | 0.004 |

⁽¹⁾ Median of the accepted data sets, each set being obtained by a different laboratory and/or a different method of determination.

⁽²⁾ Estimated expanded uncertainty with a coverage factor $k=2$, corresponding to a level of confidence of approximately 95%, as defined in the JCGM100:2008 Evaluation of measurement data - Guide to the expression of uncertainty in measurement [1].

Origin and preparation of the material

A sample of 200 kg of Scallop (*Pecten maximus*) was collected in December 2007 and January 2008 by scuba diving in the "Pertuis Breton", Western France. The organisms were dissected and soft tissues (the gills, mantle and digestive glands) lyophilized. The freeze dried matter was milled to a powder in a grinder and then sieved through a 250 µm sieve. The sieved material with a particle size of less than 250 µm was further homogenized.

Homogeneity was achieved by mixing the material in a stainless steel rotating homogenizer for 14 days in a clean atmosphere 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 and then sealed in plastic bags. The sample material was labelled IAEA-452.

Characterization study

Twenty laboratories from Austria, Brasil, Canada, Chile, Croatia, Finland, Greece, Italy, Israel, Macao, Poland, U.K, U.S.A., including the IAEA's Marine Environmental Studies Laboratory (MESL) in Monaco, participated in the characterization study.

Laboratories were requested to determine as many trace elements as possible by their analytical method of choice. The following methods were used: Atomic-Absorption Spectrometry (Flame and Graphite Furnace), Inductively-Coupled Optical Emission Spectrometry, Inductively-Coupled Mass Spectrometry, Neutron-Activation Analysis and Atomic Fluorescence Spectrometry.

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 median concentrations for the sets of individual data were chosen as the best estimate of the property values [2]. Certified values are assigned when at least 7 laboratory means were available, and at least 4 different analytical methods applied.

The details concerning all reported results as well as the criteria for certification may be found in [2, 3]. The report "Worldwide Laboratory Comparison on the Determination of Trace Elements in IAEA-452 Biota Sample", IAEA, Vienna, 2012 may be downloaded free of charge from:

[http://nucleus.iaea.org/rpst/ReferenceProducts/ReferenceMaterials/Trace Elements Methylmercury/index.htm](http://nucleus.iaea.org/rpst/ReferenceProducts/ReferenceMaterials/Trace%20Elements%20Methylmercury/index.htm)

All other documents are available free of charge upon request.

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.

Statement on metrological traceability and uncertainty of assigned values

Expanded uncertainties with a coverage factor of $k=2$, corresponding to a level of confidence of about 95%, were calculated according to the JCGM100:2008 Evaluation of measurement data - Guide to the expression of uncertainty in measurement [1].

The quantity values assigned to the IAEA-452 certified reference material are mass fractions of specified trace elements, expressed in the derived SI unit mg kg^{-1} . Evidence on metrological traceability to the SI Units of reference materials and calibrators used in the characterization process was provided by all laboratories in their reports. More details can be found in the references [2, 3].

Intended use

This Certified Reference Material is intended to be used in the development, validation and quality control of analytical methods for the determination of trace elements in marine biota samples.

Instructions for use

The reference material is supplied in 8 g units. Based on results obtained for within and between bottles homogeneity, the minimum test portion was set to be 0.2 g.

Homogeneity of the material

Between-bottle homogeneity was tested by the determination of the mass fraction of all certified elements (As, Cd, Cr, Cu, Fe, Hg, Mn, Pb, Zn and MeHg). In total, 10 bottles were selected using random stratified sampling of the whole batch. Three subsamples from each bottle were analysed for their total element mass fractions. The within bottle homogeneity was assessed by 15 replicate determinations of mass fraction of trace elements in one bottle. The final measurements were performed by Graphite Furnace Atomic Absorption Spectrometry after microwave acid digestion. The determination of the total mercury was done in solid subsamples with solid mercury analyser. The method used for homogeneity study of MeHg was based on alkaline digestion and aqueous phase ethylation, followed by gas chromatography separation, thermal desorption of Hg species and Atomic Fluorescence Spectrometry detection. All analytical procedures used for homogeneity studies were previously validated in the IAEA-MESL.

The analysis 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 were attained and the uncertainty due to between and within bottles heterogeneity was within acceptable limits.

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 (± 2)°C until constant mass and was found to be approximately 6.5 (± 0.8)% ($k=2$). Since the moisture content can vary with ambient humidity and temperature, it is recommended to check it prior to analysis and to correct 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. 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 reference material is **March 2012**. The expiry date is **March 2017**. 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 31:2000

The content of this IAEA Reference Sheet is in compliance with the ISO Guide 31:2000: Reference materials – Content of certificates and labels [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-452, 'Trace elements and methyl mercury in biota', 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 may be found in the IAEA's Reference Materials online catalogue:

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

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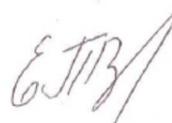
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- [3] VASILEVA, E., AZEMARD, S., OH, J., BUSTAMANTE, P., BETTI, M., Certification for trace elements and methyl mercury mass fractions in IAEA-452 scallop (*Pecten maximus*) sample, Accred. Qual. Assur. **16** 2011 439-447.
- [4] INTERNATIONAL ORGANIZATION FOR STANDARDIZATION (ISO), Reference materials – Content of certificates and labels, ISO Guide 31: 2000, ISO, Geneva (2000).



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