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REFERENCE SHEET

REFERENCE MATERIAL

IAEA-407

TRACE ELEMENTS AND METHYLMERCURY IN FISH TISSUE

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Recommended values: Trace Elements
(Based on dry weight)

Element	Concentration [1] (mg kg^{-1})	Std Deviation [2] (mg kg^{-1})	95% Confidence Interval [3] (mg kg^{-1})	N [4]
Aluminium	**13.8**	**3.7**	**12.4 – 15.2**	**31**
Arsenic	**12.6**	**1.2**	**12.3 – 12.9**	**60**
Bromine	**94**	**9**	**86 – 102**	**7**
Cadmium	**0.189**	**0.019**	**0.185 – 0.193**	**75**
Cobalt	**0.10**	**0.02**	**0.09 – 0.11**	**27**
Chromium	**0.73**	**0.22**	**0.67 – 0.79**	**65**
Copper	**3.28**	**0.40**	**3.20 – 3.36**	**90**
Iron	**146**	**14**	**143 – 149**	**71**
Mercury	**0.222**	**0.024**	**0.216 – 0.228**	**74**
Lithium	**0.685**	**0.094**	**0.62 – 0.74**	**13**
Manganese	**3.52**	**0.32**	**3.44 – 3.60**	**64**
Nickel	**0.60**	**0.18**	**0.55 – 0.65**	**49**
Lead	**0.12**	**0.06**	**0.10 – 0.14**	**61**
Rubidium	**2.86**	**0.40**	**2.45 – 3.27**	**6**
Antimony	**0.011**	**0.002**	**0.010 – 0.012**	**12**
Selenium	**2.83**	**0.38**	**2.70 – 2.96**	**38**
Strontium	**130**	**11**	**125 – 135**	**26**
Vanadium	**1.43**	**0.20**	**1.34 – 1.52**	**22**
Zinc	**67.1**	**3.8**	**66.3 – 67.9**	**93**
Me-Hg [5]	**0.200**	**0.021**	**0.188 – 0.212**	**16**

[1] Mean values expressed on a dry-weight basis.

[2] 1 standard deviation of the mean.

[3] 95% Confidence Interval, μ, defined as $\bar{x} \pm t(\frac{s}{\sqrt{N}})$, where *t* is the Student's *t* value.

[4] Number of accepted laboratory means which were used to calculate the Recommended values and confidence intervals about the mean values.

[5] Concentration reported as mg Hg kg^{-1}

Recommended values: Matrix and Minor Constituents
(Based on dry weight)

Element	Concentration [1] ($g\ kg^{-1}$)	Std Deviation [2] ($g\ kg^{-1}$)	95% Confidence Interval [3] ($g\ kg^{-1}$)	N [4]
Calcium	**27.0**	**1.8**	**25.7 – 28.3**	**9**
Potassium	**13.1**	**1.2**	**12.2 – 14.0**	**9**
Magnesium	**2.72**	**0.14**	**2.58 – 2.86**	**7**
Sodium	**13.1**	**0.6**	**12.4 – 13.8**	**5**

Information values
(Based on dry weight)

Element	Concentration [1] ($mg\ kg^{-1}$)	Std Deviation [2] ($mg\ kg^{-1}$)	95% Confidence Interval [3] ($mg\ kg^{-1}$)	N [4]
Silver	**0.037**	**0.002**	**0.033 - 0.041**	**3**
Tin	**0.10**	**0.05**	**0.06 - 0.13**	**12**

[1] Mean values expressed on a dry-weight basis.

[2] 1 standard deviation of the mean.

[3] 95% Confidence Interval, μ, defined as $\bar{x} \pm t(\frac{s}{\sqrt{N}})$, where t is the Student's t value.

[4] Number of accepted laboratory means which were used to calculate the reference values and confidence intervals about the mean values.

Establishment of reference values

The values listed above were established on the basis of results submitted by laboratories which had participated in an international intercomparison exercise organized in 2000. The details concerning the criteria for qualification as recommended or information value can be found in the "Report on the world-wide intercomparison exercise for the determination of trace elements and methylmercury in fish homogenate IAEA-407" [1]. This report is available free of charge upon request.

Intended use

This material is intended to be used as a reference material for the measurement of trace elements and methylmercury (MeHg) in fish tissue samples. It can also be used as a quality control material for the assessment of analytical procedures, in the elaboration and validation of analytical methods, and for educational purposes.

Origin and preparation of the material

A large quantity of whole fish was collected in 1999 from the North Sea for use as an intercomparison material. Most of what was collected was herring, but the sample material also contained capelan and anchovy. It was deep-frozen, freeze-dried, triple-extracted with acetone to remove fat, and then ground and sieved. The particle size fraction <250 μm was homogenized by mixing in a rotating glass bottle for 15 days. After checking for the

homogeneity of the sample material (see below), aliquots of about 35 g were packed into cleaned brown borosilicate glass bottles with Teflon lined screw caps and sealed in plastic bags. Approximately 500 bottles were produced.

Homogeneity

Extensive homogeneity tests were carried out on this material in order to ensure its suitability as an intercomparison sample. A homogeneity pre-test was conducted before bottling the sample material. The between-bottle homogeneity was tested by the determination of the concentration of some typical elements (Cd, Cr, Cu, Fe, Mn, Zn) on sample intakes of 0.2 g taken from 10 bottles which were taken directly from the bulk material, specifically for the purpose of this pre-test. The within-bottle homogeneity was assessed by 10 replicate determinations on the re-homogenized content of one bottle. The uncertainty of the analytical methods was assessed for each element by 5 replicate measurements on one digest solution.

Both the within-bottle and between-bottle CV's observed for Cr and Fe were significantly higher than the method CV's for these elements -- a clear indication that the sample material was not entirely homogeneous, at least with respect to certain constituents. An F-test at a significance level of 0.05 was performed for the different metals and did not reveal significant differences between the within- and between-bottle variances, indicating that the heterogeneity observed was relatively consistent, and independent of how the material was distributed.

In spite of the relatively large variations observed for Cr and Fe, the sample material was bottled and a final homogeneity test performed on 20 random bottles for Cd and Cu only. The variances observed for these elements compared well with the variances found in the pre-test. It was concluded that while not entirely homogeneous for all elements of interest, the material was homogeneous for most elements and sufficiently so for the others at an analytical portion of 200 mg and above; it is therefore suitable for use as an intercomparison sample [1].

Stability tests

The stability of several trace metals was tested to determine the suitability of this material as a candidate CRM. Five bottles of the IAEA-407 material were stored in the dark at +20 °C, –20 °C and +60 °C, respectively, over a period of 18 months (starting in September 2000) and the measurement of total Hg, Cu, Fe, Mn and Zn was performed at regular intervals during the storage period. On the basis of these results, it was concluded that no instability of the material could be demonstrated [1].

Dry weight determination

The average moisture content of the lyophilized sample after bottling, determined by drying to a constant weight at 105°C, was found to be 6 %. Since the moisture content can vary with the ambient humidity and temperature, it is recommended that the water content of this material be determined in a separate subsample (not used for analysis) by drying to a constant weight (~24 hours) at 105°C just prior to analysis. Final results should always be reported on a dry weight basis.

Instructions for use

The recommended minimum sample size for analysis is 200 mg. Analysts are reminded to take appropriate precautions in order to avoid contaminating the remaining material in the

bottle. The bottle should be thoroughly mixed by shaking before use and tightly resealed immediately after use. The material should be stored in the dark and kept below 25 ºC.

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.

Reference

[1] Wyse, E. J., S. Azemard and S. J. de Mora, 2003. Report on the world-wide intercomparison exercise for the determination of trace elements and methylmercury in fish homogenate IAEA-407. IAEA/AL/144, IAEA/MEL/72, IAEA, pp. 94.