



International Atomic Energy Agency
Analytical Quality Control Services
Wagramer Strasse 5, P.O. Box 100, A-1400 Vienna,
Austria

REFERENCE SHEET

REFERENCE MATERIAL

IAEA-158

TRACE ELEMENTS AND METHYLMERCURY IN MARINE SEDIMENT

Date of issue: 31 March 2008

Recommended values: Trace Elements

(Based on dry weight)

Element	Concentration ¹ (mg kg ⁻¹)	Std Deviation ² (mg kg ⁻¹)	n ³
Silver	0.180	0.033	5
Arsenic	11.5	1.2	21
Cadmium	0.372	0.039	25
Cerium	61.1	5.4	5
Cobalt	9.2	1.1	17
Chromium	74.4	5.8	23
Caesium	3.73	0.34	6
Copper	48.3	4.2	44
Europium	1.079	0.061	5
Lanthanum	30.2	2.2	7
Manganese	356	24	23
Nickel	30.3	2.9	22
Lead	39.6	4.7	29
Rubidium	82	10	7
Antimony	1.34	0.18	8
Samarium	4.94	0.32	6
Tin	5.84	0.82	5
Strontium	473	25	8
Uranium	2.42	0.28	5
Vanadium	73.0	3.7	9
Zinc	140.6	9.5	41

¹ Mean values expressed on a dry-weight basis.

² 1 standard deviation of the mean.

³ Number of Laboratory means from the total which were used by Cofino model to calculate the Recommended values.

Recommended values: Matrix and Minor Constituents

(Based on dry weight)

Element	Concentration ¹ (g kg⁻¹)	Std Deviation ² (g kg⁻¹)	n ³
Aluminium	51.8	3.4	7
Calcium	64.9	5.8	5
Iron	26.3	1.4	24
Potassium	20.0	1.6	7
Sodium	23.8	1.0	7

Information values

(Based on dry weight)

Element	Concentration ¹ (mg kg⁻¹)	Std Deviation ² (mg kg⁻¹)	n ³
Barium	1028	46	4
Bromine	224	15	4
Hafnium	6.23	0.4	5
Mercury	0.132	0.014	24
Lithium	32.5	3.7	4
Lutetium	0.306	0.03	5
MeHg⁴	0.00141	0.0004	10
Magnesium	10390	960	4
Molybdenum	4.87	0.8	5
Scandium	8.32	0.39	5
Tantalum	0.97	0.12	5
Terbium	0.63	0.097	4
Thorium	8.89	0.58	5
Titanium	3490	170	4
Ytterbium	2.08	0.18	4

¹ Mean values expressed on a dry-weight basis.

² 1 standard deviation of the mean.

³ Number of Laboratory means from the total which were used by Cofino model to calculate Recommended and Information values.

⁴ Concentration reported as mg Hg kg⁻¹

Determination of reference values

The values listed above were established on the basis of results submitted by laboratories that had participated in an international intercomparison exercise, organized in 2006. 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 marine sediment IAEA-158” [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 sediment 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

In November 2004, a large quantity of marine sediment was collected from Kilbrannan Sound, south east of the island of Arran, in the Clyde River estuary, Scotland, UK. The material was collected and supplied to the IAEA through a collaboration with the QUASIMEME Laboratory Performance Studies Programme. The material was freeze dried by QUASIMEME and sent to IAEA MEL for further processing and bottling. The dried, material was hand sieved (315 μm) by MESL staff. Approximately 70% of particles had sizes below 100 μm . Aliquots of about 25 g were packed into glass bottles with polyethylene caps and sealed in plastic bags. Approximately five hundred units of the material were prepared. The homogeneity of this material for trace elements was tested using a standard protocol and found to be satisfactory.

Homogeneity

Extensive homogeneity tests were carried out on this material in order to ensure its suitability as an intercomparison sample. The between-bottle homogeneity was tested by the determination of the concentration of some typical elements (Cd, Cu, Fe, Pb, Mn, Hg and Zn) on sample intakes of 0.25 g taken from 6 bottles. The within-bottle homogeneity was assessed by 6 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 or by 5 replicate measurements of a certified reference for Hg.

An F-test at a significance level of 0.05 was performed for the different metals and did not reveal significant differences between within- and between-bottle variances, indicating that the heterogeneity observed was relatively consistent, and independent of how the material was distributed. Results of Hg showed unhomogeneity of the material linked to “outliers” results; as a consequence Hg was classified as an information value. As no extensive study of homogeneity was performed for MeHg, by precaution the reference value is also classified as information value.

It was concluded that the material was homogeneous for the elements tested (at the exception of Hg) at an analytical portion of 250 mg and above; it is therefore suitable for use as an intercomparison sample [1].

Instructions for use

The recommended minimum sample size for analysis is 250 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 °C. Since the moisture content can vary with the ambient humidity and temperature, the water content of this material must be determined in a separate sub-sample (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. Failure to compensate for moisture content will lead to underestimation of the results.

It appears evident during data treatment that some elements (Al, Cr, Fe, Mn, Sr and V) could be partially solubilized. It is necessary to use hydrofluoric acid to achieved accurate data for Al and Sr. For Cr, Fe, Mn and V strong acid digestion process are recommended.

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] M. J. Campbell, S. Azemard and J. Oh, 2008. Report on the World-wide intercomparison exercise on the determination of trace elements in IAEA-158 marine sediment. IAEA/AL/187, IAEA/MEL/82, IAEA, Austria.