



REFERENCE SHEET

REFERENCE MATERIAL

IAEA-405

TRACE ELEMENTS AND METHYLMERCURY IN ESTUARINE SEDIMENT

Date of issue: 1 August 2000

Recommended Values
(Based on dry weight)

Analyte	Recommended Value [mg/kg]	95% Confidence Interval [mg/kg]	N*
As	23.6	22.9 – 24.3	47
Cd	0.73	0.68 – 0.78	63
Co	13.7	13.0 – 14.4	50
Cr	84	80 – 88	63
Cu	47.7	46.5 – 48.9	80
Fe	37400	36700 – 38100	64
Hg	0.81	0.77 – 0.85	60
Li	72	65 – 79	25
Mg	12300	11400 – 13200	13
Mn	495	484 – 506	52
Ni	32.5	31.1 – 33.9	61
Pb	74.8	72.6 – 77.0	74
Sb	1.81	1.62 – 2.00	21
Se	0.44	0.32 – 0.56	12
Sn	7.6	6.3 – 8.9	17
V	95	90 – 100	29
Zn	279	272 – 286	87
MeHg [§]	0.00549	0.00496 – 0.00602	12

* Number of accepted laboratory results which were used to calculate the recommended values and confidence intervals about the mean value

§ As inorganic Hg

Information Values
(Based on dry weight)

Analyte	Information Value [mg/kg]	95% Confidence Interval [mg/kg]	N*
Al	77900	72700 – 83100	37
Br	85	60 – 110	4
Cs	12.5	10.4 – 14.6	4
Eu	1.25	0.89 – 1.61	5
Hf	5.80	4.93 – 6.67	3
K	24900	17700 – 32100	5
La	40.4	33.1 – 47.7	5
Lu	0.468	0.283 – 0.653	3
Sc	13.52	11.53 – 15.51	3
Sm	6.86	6.50 – 7.22	4
Sr	118	104 – 132	28
Tb	0.93	0.50 – 1.36	3
Th	14.3	12.2 – 16.4	5
U	3.01	1.86 – 4.16	5
Yb	3.04	2.19 – 3.89	4

* Number of accepted laboratory results which were used to calculate the information values and confidence intervals about the mean value

The values listed above were established on the basis of statistically and technically valid results submitted by laboratories which had participated in an international intercomparison exercise organized in 1998. The details concerning the criteria for qualification as a recommended or information value can be found in the report (IAEA/AL/127; IAEA/MEL/70), “Report on the World-wide Intercomparison Exercise for the Determination of Trace Elements and Methylmercury in Estuarine Sediment IAEA-405” [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 coastal sediments. 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 sediment was collected in 1998 from the intertidal mudflats of the Tagus estuary (Portugal) for use as an intercomparison material. It was deep-frozen, freeze-dried, ground and sieved. The sediment fraction of particle size less than 150 µm was further homogenized by mixing in a stainless steel rotating drum for two weeks. After checking for the homogeneity of the sample material (see below), aliquots of about 35g were packed into cleaned brown borosilicate glass bottles with Teflon lined screw caps and sealed in plastic bags. A total of 530 bottles was produced.

Homogeneity

Extensive homogeneity tests were carried out on this material in order to ensure its suitability as an intercomparison sample. A preliminary test was performed before final bottling and sample dispatch and did not detect any inhomogeneity in the material. A final homogeneity test was conducted after completion of the bottling of sample material. The between-bottle homogeneity was tested by the determination of the concentration of some typical elements (Cu, Fe, Mn, Zn) on sample intakes of 100 mg and 200 mg taken from 15 bottles which were set aside at regular intervals during the whole period of bottling. The within-bottle homogeneity was assessed by 15 replicate determinations on the re-homogenized content of one bottle. A 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 for 100 mg intakes. On the basis of these results, no inhomogeneities in the material were suspected. It was concluded that the material is homogeneous at an analytical portion of 100 mg and above for trace elements and, therefore, suitable for use as an intercomparison sample [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 2.5 %. Since the moisture content can vary with the ambient humidity and temperature, it was 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.

Stability of the material

The stability of several trace metals was tested to determine the suitability of this material as a candidate CRM. Five bottles of the IAEA-405 material were stored in the dark at +20 °C, -20 °C and +60 °C, respectively, over a period of 17 months (starting in September 1998) 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].

Instructions for use

The recommended minimum sample size for analysis is 100 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.

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] Coquery M., Azemard S. and de Mora S. J., Report on the World-wide Intercomparison Exercise for the Determination of Trace Elements and Methylmercury in Estuarine Sediment IAEA-405, IAEA/AL/127 (IAEA/MEL/70), IAEA, Monaco, 2000.

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Analytical Quality Control Services (AQCS)
Agency's Laboratories, Seibersdorf
International Atomic Energy Agency
P.O. Box 100
A-1400 Vienna, Austria

Prepared by

M. Coquery, S. Azemard, S. J. de Mora