Certified Reference Material

National Research Council Canada | Conseil national de recherches Canada

HISS-1, MESS-3, PACS-2

Marine Sediment Reference Materials for Trace Metals and other Constituents

The following tables show those constituents for which certified and information values have been established. Certified values are based on the results of determinations by at least two independent methods of analysis. The uncertainties represent 95% confidence limits for an individual sub-sample of 250 mg or greater. The uncertainties in the certified values of the butyltins are based on the expanded uncertainties.**

Trace Metals

(milligrams per kilogram)

	HISS-1	MESS-3	PACS-2
Antimony	(0.13)*	1.02 ± 0.09	11.3 ± 2.6
Arsenic	0.801 ± 0.099	21.2 ± 1.1	26.2 ± 1.5
Beryllium	0.129 ± 0.023	2.30 ± 0.12	1.0 ± 0.2
Cadmium	0.024 ± 0.009	0.24 ± 0.01	2.11 ± 0.15
Chromium	30.0 ± 6.8†	105 ± 4	90.7 ± 4.6
Cobalt	(0.65)*	14.4 ± 2.0	11.5 ± 0.3
Copper	2.29 ± 0.37	33.9 ± 1.6	310 ± 12
Lead	3.13 ± 0.40	21.1 ± 0.7	183 ± 8
Lithium	2.83 ± 0.54	73.6 ± 5.2	32.2 ± 2.0
Manganese	66.1 ± 4.2	324 ± 12	440 ± 19
Mercury	(0.01)*	0.091± 0.009	3.04 ± 0.20
Molybdenum	(0.13)*	2.78 ± 0.07	5.43 ± 0.28
Nickel	2.16 ± 0.29	46.9 ± 2.2	39.5 ± 2.3
Selenium	0.050 ± 0.007	0.72 ± 0.05	0.92 ± 0.22
Silver	0.016 ± 0.002	0.18 ± 0.02	1.22 ± 0.14
Strontium	96.9 ± 11.2	129 ± 11	276 ± 30
Thallium	(0.06)*	0.90 ± 0.06	(0.6)*
Tin	(0.11)*	2.50 ± 0.52	19.8 ± 2.5
Uranium	(0.26)*	(4)*	(3.)*
Vanadium	6.80 ± 0.78	243 ± 10	133 ± 5
Zinc	4.94 ± 0.79	159 ± 8	364 ± 23
Tributyltin (as Sn)**	—-	—-	0.890± 0.105
Dibutyltin (as Sn)**	—-	—	1.047± 0.064
Monobutyltin (as Sn)**	—-	—-	(0.6)*

*information value only
† see page 3
** a separate certificate for the butyltins is available.

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Matrix and Minor Constituents - Percent

		HISS-1	MESS-3	PACS-2
Al	(f,i,n,x)	0.73 ± 0.05	8.59 ± 0.23	6.62 ± 0.32
C	(e)	—-	(2)*	(3.3)*
Ca	(f,i,n,x)	1.14 ± 0.10	1.47 ± 0.06	1.96 ± 0.18
Cl	(n,x)	(0.35)*	—-	(3.)*
Fe	(f,i,n,x)	0.246 ± 0.009	4.34 ± 0.11	4.09 ± 0.06
K	(f,n,x)	0.332 ± 0.013	(2.6)*	1.24 ± 0.05
Mg	(f,i,x)	0.075 ± 0.016	(1.6)*	1.47 ± 0.13
Na	(f,i,n)	0.373 ± 0.026	(1.6)*	3.45 ± 0.17
P	(i,x)	—-	(0.12)*	0.096 ± 0.004
S	(i,x)	—-	(0.19)*	1.29 ± 0.13
Si	(i,x)	(44.)*	(27.)*	(28.)*
Ti	(f,i,n,x)	0.076 ± 0.004	0.44 ± 0.06	0.443 ± 0.032

Methods of Determination for Trace Metals

Antimony (b,g,i,n,q)
Arsenic (b,g,h,i,n,x)
Beryllium (b,g,i)
Cadmium (b,g,i,q)
Chromium (b,g,i,n,q,x)
Cobalt (b,g,i,n,x)
Copper (b,g,i,n,q,x)
Lead (b,g,i,q,x)
Lithium (b,g,i,q)
Manganese (b,g,i,n,x)
Mercury (a,c,q)
Molybdenum (g,i,q)
Nickel (b,g,i,q,x)
Selenium (b,g,h)
Silver (b,g,i,q)
Strontium (f,i,n,q,x)
Thallium (b,q)
Tin (b,g,i,q)
Uranium (q)
Vanadium (b,g,i,n,x)
Zinc (b,g,i,n,q,x)
organotin species (l,m)

Coding

a - Atomic fluorescence spectrometry
b - Inductively coupled plasma mass spectrometry (ICPMS)
c - Cold vapour atomic absorption spectrometry
e - Coulometry
f - Flame atomic absorption spectrometry
g - Graphite furnace atomic absorption spectrometry
h - Hydride generation atomic absorption spectrometry
i - Inductively coupled plasma atomic emission spectrometry
l - High-performance liquid chromatography ICPMS
m - Gas chromatography microwave induced plasma atomic emission
n - Instrumental neutron activation analysis
q - Isotope dilution inductively coupled plasma mass spectrometry
r - Infrared spectrometry
x - X-ray fluorescence spectrometry

Not all the methods listed above were applied to all three certified reference materials.

These reference materials are primarily intended for use in the calibration of procedures and the development of methods used for the analysis of marine sediments and materials with similar matrices.

Preparation of material

HISS-1 was collected from the Hibernia Shelf, off the coast of Newfoundland.
MESS-3 is from the Beaufort Sea.
PACS-2 was collected in the harbour of Esquimalt, B.C. They were all freeze dried, screened to pass a No. 120 (125 µm) screen, blended and bottled by Institute staff using the facilities of the Canada Centre for Mineral and Energy Technology in Ottawa. After bottling, the samples were radiation sterilized with a minimum dose of 2.5 Mrad by Nordion International Inc. to minimize any effects from biological activity.

Instructions for drying

Although initially free from moisture following the freeze drying, the materials, which contain sea salt, have picked up moisture during subsequent operations. They should be dried to a constant weight before use. Drying for several hours at 105EC has proved to be a relatively simple method to achieve a dry weight for most purposes.

Storage

Repeated analysis of these materials has demonstrated that elemental concentrations are stable for at least 10 years when bottles are stored well sealed, and in a cool place. Each bottle is packaged in a trilaminate foil pouch as an impermeable barrier to mercury vapour. Experiments have shown that, under conditions of high ambient levels of mercury vapour, mercury is able to penetrate the plastic cap of the bottle, thereby potentially contaminating the contents.

Storage of PACS-2 for Organotin Stability

To ensure the stability of the organotin species in PACS-2 it is necessary to store the material at a temperature of 4°C or lower.

Information values

Information values are considered less reliable than certified values because they are not based on the results of at least two independent methods, there were insufficient analyses performed or inhomogeniety is suspected. These numbers are given for information only and care should be excised not to attribute more reliability to these numbers than they warrant.

Homogeneity

Randomly selected bottles were used for the analytical determinations. Results from different bottles showed no significant differences compared to results from sub-samples within bottles. Nor was there any correlation between values obtained and bottle sequence. Thus, it is assumed that all bottles of each of these materials have essentially the same composition. One exception is Co in HISS-1 where results indicated sample inhomogeniety.

†Chromium in HISS-1

It became apparent during the certification of HISS-1 that there is a significant fraction of Cr that is not easily solubilized. The certified value of 30 mg/kg was obtained using solid sampling techniques or prolonged digestion with hydrofluoric, sulphuric and perchloric acids. Less vigorous acid dissolution techniques (including microwave heating using closed vessels at high pressure) result in Cr values between 10 and 13 mg/kg.

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Date of expiry (butyltins): September 2007

The results listed in this certificate are traceable to the SI through gravimetrically prepared standards of established purity and international measurement intercomparisons. As such, they serve as suitable reference materials for laboratory quality assurance programs, as outlined in ISO/IEC 17025. These CRMs are registered at the Bureau International des Poids et Mesures (BIPM) in Appendix C of the Comité International des Poids et Mesures database listing Calibration and Measurement Capabilities accepted by signatories to the Mutual Recognition Arrangement of the Metre Convention.

Comments, information and inquiries should be addressed to:

Dr. R.E. Sturgeon
National Research Council of Canada
Institute for National Measurement Standards
M-12, Montreal Road
Ottawa, Ontario, Canada K1A 0R6

Telephone (613) 993-2359
Facsimile (613) 993-2451
E-mail crm.inms@nrc-cnrc.gc.ca

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