



PACS-3

Marine Sediment Certified Reference Material 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 results from data generated at NRC.

The expanded uncertainty (U_{CRM}) in the certified value is equal to $U = ku_c$ where u_c is the combined standard uncertainty calculated according to the JCGM Guide [1] and k is the coverage factor. It is intended that U_{CRM} accounts for every aspect that reasonably contributes to the uncertainty of the measurement. A coverage factor of 2 was applied for all elements. The table below lists certified values for PACS-3 expressed on a dry mass basis.

Table 1a: Certified Quantity Values – Trace Metals

Element	Mass fraction, mg/kg
Antimony (a,c)	14.6 ± 2.4
Arsenic (b,c)	30.3 ± 2.3
Beryllium (b)	1.07 ± 0.12
Cadmium (a)	2.23 ± 0.13
Chromium (a,c)	91.6 ± 4.0
Copper (a,c)	327 ± 14
Lead (a,c)	188 ± 7
Lithium (a,c)	31.9 ± 5.6
Manganese (b,c)	425 ± 34
Mercury (a)	3.0 ± 0.5
Nickel (a,c)	39.9 ± 3.0
Silver (a,c)	1.37 ± 0.36
Strontium (a,c)	268 ± 12
Tin (a,c)	22.0 ± 1.5
Vanadium (b,c)	129 ± 9
Zinc (a,c)	379 ± 15

Table 1b: Certified Quantity Values – Matrix Constituents

Element	Mass fraction, g/(100 g) (%)
Aluminum (c,d)	6.58 ± 0.17
Calcium (c,d)	1.90 ± 0.10
Iron (c,d)	4.11 ± 0.11
Potassium (c,d)	1.24 ± 0.07
Magnesium (c,d)	1.47 ± 0.19
Phosphorus (c,d)	0.091 ± 0.009
Titanium (c,d)	0.44 ± 0.05

Table 1c: Certified Quantity Values – Butyltin Compounds

Compound	Mass fraction, mg/kg (as Sn)
Dibutyltin (e,f)	0.62 ± 0.04
Tributyltin (e,f)	0.41 ± 0.04

Table 2: Information Values: Trace and Matrix Constituents

Element	Mass fraction, mg/kg
Cobalt (b)	12.1
Molybdenum (a)	5.9
Thallium (a)	0.62
Uranium (a)	2.6
Element	Mass fraction, g/(100 g) (%)
Sodium (c)	3.5
Sulfur (d)	1.2
Silicon (d)	26.1

Coding

The coding refers only to the instrumental method of analyte determination.

- a - Isotope dilution inductively coupled plasma mass spectrometry
- b - Inductively coupled plasma mass spectrometry (ICPMS)
- c - Inductively coupled plasma atomic emission spectrometry
- d - X-ray fluorescence spectrometry
- e - Isotope dilution gas chromatography mass spectrometry
- f - Isotope dilution gas chromatography ICPMS

Certified Values

Certified values are considered to be those for which NRC has the highest confidence in accuracy and that all known and suspected sources of bias have been taken into account and are reflected in the stated expanded uncertainties.

Information values

Information values are non-certified values for which insufficient data are available to provide an estimate of uncertainty to permit their full certification.

Intended Use

This reference material is primarily intended for use in the calibration of procedures and the development of methods for the determination of trace and matrix constituents in marine sediments and materials with similar matrices.

Storage and Sampling

It is recommended that the material be stored in a cool, clean location. Each bottle is packaged in a trilaminate foil pouch which serves as an impermeable barrier to mercury vapour. 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. The bottle contents should be well mixed by rotation and shaking prior to use, and tightly closed immediately thereafter. Certified values are based on a minimum 0.250 g sub-sample from the bottle.

Storage of PACS-3 for Butyltin Stability

To ensure the stability of the butyltin species in PACS-3, it is necessary to store the material at a temperature of 4 °C or lower.

Instructions for drying

Although initially free from moisture following the freeze drying, the materials, which contain sea salt, have adsorbed moisture during subsequent operations. They should be dried to a constant mass before use. Drying for several hours at 105 °C is recommended as a relatively simple method to achieve a dry mass for most purposes.



Preparation of material

PACS-3 was collected from Esquimalt harbour, B.C. All were 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 25 kGy by MDS Nordion Inc. to minimize any effects from biological activity.

Stability

The predecessor CRM, PACS-2, has been periodically analyzed for more than ten years and found to be both physically and chemically stable over this time interval. We expect similar results for PACS-3. Uncertainty components for long and short term stability were considered negligible and are thus not included in the uncertainty budget.

Homogeneity

The material was tested for homogeneity at NRC. Results from sub-samples (0.250 g) were evaluated using ANOVA and included in the calculation of the certified values.

Uncertainty

Included in the overall combined uncertainty estimate (u_c) are uncertainties in the batch characterization (u_{char}) and uncertainties related to possible between-bottle variation (u_{hom}). Expressed as standard uncertainties, these components are listed in Table 3.

Table 3a: Uncertainty components for PACS-3

Element	u_c , mg/kg	u_{char} , mg/kg	u_{hom} , mg/kg
Sb	1.2	1.1	0.4
As	1.1	1.0	0.6
Be	0.06	0.02	0.05
Cd	0.07	0.06	0.03
Cr	2.0	1.5	1.4
Cu	7	6	3
Pb	3	2	3
Li	2.8	2.7	0.6
Mn	17	17	3
Hg	0.23	0.22	0.09
Ni	1.5	1.3	0.8
Ag	0.18	0.18	0.04
Sr	6	2	6
Sn	0.7	0.3	0.6
V	4	4	1
Zn	8	7	4
Dibutyltin	0.02	0.02	0.01
Tributyltin	0.02	0.01	0.01

Table 3b: Uncertainty components for PACS-3

Element	$u_{c,}$ g/(100 g) (%)	$u_{char,}$ g/(100 g) (%)	$u_{hom,}$ g/(100 g) (%)
Al	0.09	0.08	0.03
Ca	0.05	0.05	0.01
Fe	0.06	0.05	0.03
K	0.04	0.03	0.01
Mg	0.10	0.10	0.01
P	0.0045	0.0045	0.0002
Ti	0.02	0.01	0.02

Metrological Traceability

Results presented 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. This CRM is 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.

Accreditation

The Chemical Metrology laboratory is compliant to ISO 17025 and ISO Guide 34, with approval by The Inter-American Metrology System (SIM). The certificate of approval is available upon request.

Updates

Users should ensure that the certificate they have is current. Our web site at <http://www.nrc.ca/crm> will contain any new information.

References

1. Evaluation of measurement data – Guide to the expression of uncertainty in measurement JCGM 100:2008.

Acknowledgements

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