National Institute of Advanced Industrial Science and Technology

National Metrology Institute of Japan

Reference Material Certificate

NMIJ CRM 7302-a
No. +++

Trace Elements in Marine Sediment

This certified reference material (CRM) is produced in accordance with the NMIJ's management system and is in compliance with ISO 17034 and ISO/IEC 17025. This CRM is intended for use in controlling the precision of analysis, and validating of analytical methods and instruments during the analysis of trace elements in sediment or similar matrices.

Certified Values

The certified values for 14 elements in this CRM are given in the table below. They are expressed in mass fractions after dry mass correction. The drying instruction is described in this certificate. The uncertainty of the certified value is the half-width of the expanded uncertainty interval calculated using a coverage factor (k) of 2, which gives a level of confidence of approximately 95 %.

Element	Certified value, Mass fraction (mg/kg)	Expanded uncertainty, Mass fraction (mg/kg)	Analytical method (see below*)
Sb	1.22	0.05	1,2
As	22.1	1.4	2,4,5
Cd	1.32	0.04	1,2,4
Cr	145	6	1,2,3,4
Co	12.4	1.5	2,3,4
Cu	57.8	2.3	1,2,3,4
Pb	82.7	3.8	1,2,3,4
Hg	0.52	0.03	1,2,6
Mo	1.98	0.24	1,2
Ni	25.8	1.2	1,2,3,4
Se	0.61	0.07	1,2,5
Ag	0.49	0.02	1,2
Sn	18.5	0.8	1,2
Zn	401	16	1,2,3

*Analytical methods;

1) Isotope dilution-inductively coupled plasma mass spectrometry (ID-ICP-MS)
2) ICP-MS
3) ICP optical emission spectrometry (ICP-OES)
4) Graphite furnace atomic absorption spectrometry
5) High resolution ICP-MS
6) Gold amalgam trap AAS

Analysis

The certified values of this CRM are the weighted means of results from two or more analytical methods carried out at NMIJ:

(1) ID-ICP-MS (primary method) and one or more reference methods
(2) Three or more reference methods

The expanded uncertainty in each certified value is equal to $U = ku_c$, where u_c is the combined standard uncertainty derived from (a)the analytical results, (b)the method-to-method variance, (c)the sample homogeneity, (d)the standard solution, with coverage factor (k =2) corresponding to an interval of 95 % confidence.

Metrological Traceability

The certified values, except for Ag and Se, were determined with JCSS (Japan Calibration Service System) standard solutions and the certified values of Ag and Se were determined with NIST SRM 3151 and SRM 3149, respectively. Thus, the certified values are traceable to the International System of Units (SI).

Mutual Recognition Arrangement under Meter Convention

This certificate is consistent with the calibration and measurement capabilities (CMCs) that are included in Appendix C of the Mutual Recognition Arrangement (MRA) drawn up by the International Committee for Weights and Measures (CIPM). Under the MRA, all participating institutes recognize the validity of each other's calibration and measurement certificates for the quantities, ranges and measurement uncertainties specified in Appendix C (as for Appendix C of MRA, see http://kcdb.bipm.org/AppendixC/default.asp).

Expiration of Certification

This certificate is valid until March 31, 2021, provided that the material remains unopened and is stored in accordance with the instructions given in this certificate.

Sample Form

This CRM is in the form of a brown powder, prepared from natural marine sediment. This CRM of 60 g in net volume is kept in an amber glass bottle.

Homogeneity

The homogeneity was determined by analyzing 10 bottles hierarchical-randomly selected from 1000 bottles. The trace elements were determined by ICP-MS or ICP-OES. The homogeneity is reflected to the uncertainties of the certified values.

Instructions for Storage

This CRM should be stored at a temperature between 5 °C and 35 °C in a clean place and shielded from light.

Instructions for Use

(1) Before taking the sample, the bottle is gently shaken by a hand at least 5 times. Sample amount over 0.1 g is recommended for quantification except Cr. In the case of Cr, sample amount over 0.3 g is recommended for quantification.
(2) The certified values in this CRM are given on a dry-mass basis. Moisture content should be assessed by taking a portion of the material (ca. 1 g) and drying it in an oven at 110 °C for 6 h. The dry mass correction factor at the certification was found to be 0.97.

Precautions for Handling

Wear protective mask, gloves and other protective equipment is recommended during handling. The handling, storage and disposal of this CRM must be performed in accordance with all applicable laws. Refer to the safety data sheet (SDS) on this CRM before use.

Preparation

This CRM was prepared from natural marine sediment. The sediment was collected at a bay located near an industrial area in Japan, and then air-dried, sieved (104 μm), homogenized, bottled (60 g each) and finally radiation sterilized (20 kGy).

Technical Information

The mass fractions of 12 elements in this CRM are given in the table below as information.

Element	Information value, Mass fraction (%)	Analytical method (see above*)	Element	Information value, Mass fraction (mg/kg)	Analytical method (see above*)
Al	7.3	3	Mn	710	3,4
Ca	4.2	3	P	650	3
Fe	5.4	3	Rb	74	2
Mg	1.2	3	Sr	330	2,3
K	1.6	3	V	166	3
Na	1.9	3			
Ti	0.42	2,3			

NMIJ Analysts

The technical manager for this CRM is KURAHASHI M., production manager is TAKATSU A., and the analysts are INAGAKI K., KUROIWA T., NAKAMA A., EYAMA S., and TAKATSU A.

Collaborating Laboratory for Sample Preparation

Environmental Technology Service Co., Ltd., Japan

Information

If substantive technical changes occur that affect the certification before the expiration of this certificate, NMIJ will notify the registered customer. Customer registration on the NMIJ Website (given below) will facilitate notification. Technical reports regarding this CRM can be obtained from the contact details given below.

Reproduction of Certificate

In reproducing this certificate, it should be clearly indicated that the document is a copy.

April 1, 2020

ISHIMURA Kazuhiko
President
National Institute of Advanced Industrial Science and Technology

If you have any questions about this CRM, please contact:
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Revision history
November 30, 2010: Expiration date was extended to March 31, 2021 from March 31, 2012.
April 1, 2015: “Metrology Management Center” was renamed to “Center for Quality Management of Metrology.”