

National Institute of Advanced Industrial Science and Technology

## National Metrology Institute of Japan



## Reference Material Certificate

NMIJ CRM 7303-a

No. +++



## Trace Elements in Lake 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 of in controlling the precision of analysis, and validating 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. The drying instruction is described in this certificate. The uncertainty of the certified value is the expanded uncertainty obtained by multiplying the combined standard uncertainty by a coverage factor ( $k$ ) of 2, and it is the half-width of an interval estimated to have a level of confidence of approximately 95 %.

Elements	Certified value Mass fraction (mg/kg)	Expanded uncertainty Mass fraction (mg/kg)	Analytical methods (see below*)
Sb	0.69	0.02	1,2
As	8.6	1.0	2,3,4,5
Cd	0.342	0.017	1,2,5
Cr	39.1	2.8	1,2,4,5
Co	11.1	1.1	2,4,5
Cu	23.1	3.1	1,2,4,5
Pb	31.3	1.1	1,2,4,5
Hg	0.067	0.006	1,2,6
Mo	0.96	0.07	1,2
Ni	21.8	2.5	1,2,4,5
Se	0.24	0.04	1,2,3
Ag	0.098	0.004	1,2
Sn	4.21	0.13	1,2
Zn	107	5	1,2,4

**\*Analytical methods**

- 1) Isotope dilution-inductively coupled plasma mass spectrometry (ID-ICP-MS)
- 2) ICP-MS with quadrupole system
- 3) ICP-MS with double-focusing system
- 4) ICP optical emission spectrometry (ICP-OES)
- 5) Graphite furnace atomic absorption spectrometry (GFAAS)
- 6) Gold amalgam trap AAS

**Analysis**

The certified value of this CRM was weighted mean of the results of the following analytical methods:

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

### Metrological Traceability

The certified values, except for Mo, Sn, Ag and Se, were determined with JCSS (Japan Calibration Service System) standard solutions. The certified values of Mo and Sn were determined with NMIJ standard solutions, which were defined by a primary method of measurement. The certified values of Ag and Se were determined with NIST SRM 3151 and SRM 3149, respectively. The certified value, therefore, is traceable to the International System of Units (SI).

### Mutual Recognition Arrangement under Metre Convention

The certified values of this CRM are recognized for international equivalence based on the Mutual Recognition Arrangement under the Metre Convention (CIPM MRA). The calibration measurement capabilities (CMC) of NMIJ related to this CRM are registered in the Key Comparison Database (KCDB) (see <https://www.bipm.org/kcdb/>) of the International Bureau of Weights and Measures (BIPM).

### Expiration of Certification

This certificate is valid for one year from the date of shipment, provided that this CRM is stored in accordance with the instructions given in this certificate.

### Description of the Material

This CRM is in the form of a brown powder, prepared from natural lake sediment. This CRM was packaged in amber glass bottles (60 g each).

### Instructions for Storage

This CRM should be stored at temperatures of 5 °C to 35 °C in a clean place and protected from light.

### Instructions for Use

(1) Sample amount for analysis

Sample amount of more than 0.1 g per analysis is recommended.

(2) Determination of moisture

The certified values and reference values are expressed on a dry-mass basis. Users should determine the moisture content at the time of the analysis to correct the analytical results to a dry-mass basis. Drying should be made by the following instructions. Note that the below method may result in the decomposition or loss of the analyte, and therefore, a separate sample should be used. Moisture content of CRM 7303-a was determined at NMIJ to be approximately 3%.

① Weigh 1 g of sample and oven drying at 110°C for 6 h.

② Weigh the sample after cooling in a desiccator with silica gel. The mass difference is taken to be the moisture content.

### Precautions for Handling

Wear a mask, gloves, and other personal protective equipment when 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 lake sediment. The sediment was collected at a lake located near an industrial area in Japan, and air-dried, sieved (104 µm), homogenized, bottled (60 g each) and finally radiation sterilized (20 kGy). The sample collection was conducted by Shimazu Techno-Research, Inc., Japan, and the following preparation was conducted by Environmental Technology Service Co., Ltd., Japan.

Date of Shipment: Xxxxx xx, 20xx

7303a00-030324-230320

### 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	Element	Information value Mass fraction (mg/kg)	Analytical method
Al	7.8	ICP-OES	Mn	690	ICP-OES
Ca	0.71	ICP-OES	P	390	ICP-OES
Fe	2.9	ICP-OES	Sr	110	ICP-OES
Mg	0.59	ICP-OES			
K	2.5	ICP-OES			
Na	1.4	ICP-OES			
Ti	0.37	ICP-OES			

### NMIJ Analysts

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

### Information

If substantive technical changes occur that affect the certification before the expiration of this certificate, NMIJ will notify the registered customers. 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: The limit of validity of the certificate was extended from “March 31, 2012” to “March 31, 2021.”  
“Reference Values” was changed to “Technical Information.”  
The description on “Mutual Recognition Arrangement under Meter Convention” was added.  
April 1, 2015: “Metrology Management Center” was renamed to “Center for Quality Management of Metrology.”  
May 21, 2020: The description in “Expiration of Certification” was changed to “one year from the date of shipment.”