

National Institute of Advanced Industrial Science and Technology

National Metrology Institute of Japan



Reference Material Certificate

NMIJ CRM 7405-a
No. +++Trace Elements and Arsenic Compounds in Seaweed (*Hijiki*)

This certified reference material (CRM) was produced in accordance with the NMIJ's management system and in compliance with ISO GUIDE 34:2000 and ISO/IEC 17025:2005. This CRM is intended for use in the evaluation and validation of analytical methods and instruments used for the determination of elements listed below and arsenate in *Hijiki* seaweed or similar matrices.

Certified Values

The certified values for 18 elements and water-soluble arsenate extracted using water in this CRM are given in the tables below. They are expressed in mass fractions after correcting their dry mass. The drying method 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 (g/kg)	Expanded uncertainty, Mass fraction (g/kg)	Analytical method*
Na	16.2	0.2	4, 6, 7
K	47.5	0.7	4, 6, 7
Mg	6.79	0.10	1, 4, 6
Ca	15.2	0.3	1, 4, 6, 7
Sr	1.47	0.04	1, 2, 4, 5
P	1.01	0.03	1, 3, 4

Element	Certified value, Mass fraction (mg/kg)	Expanded uncertainty, Mass fraction (mg/kg)	Analytical method*
Al	147	7	1, 4, 5
As	35.8	0.9	1, 3, 4, 5
Ba	14.6	0.3	1, 2
Cd	0.79	0.02	1, 2, 5
Co	1.07	0.06	1, 3, 4, 5
Cr	3.4	0.1	2, 3, 4
Cu	1.55	0.07	1, 2, 4, 5
Fe	311	11	1, 2, 4, 5
Mn	14.1	0.7	1, 4, 5
Ni	2.2	0.1	1, 2, 4
Pb	0.43	0.03	1, 2, 5
Zn	13.4	0.5	1, 2, 4

*Analytical methods:

- 1) Inductively coupled plasma mass spectrometry (ICP-MS)
- 2) Isotope dilution-ICP-MS

- 3) High-resolution ICP-MS
- 4) Inductively coupled plasma optical emission spectrometry (ICP-OES)
- 5) Graphite furnace atomic absorption spectrometry
- 6) Flame atomic absorption spectrometry
- 7) Flame photometry

(Microwave acid digestion was performed for sample pretreatment)

Compound	Certified value, Mass fraction (mg/kg)	Expanded uncertainty, Mass fraction (mg/kg)
Arsenate (as As)	10.1	0.5

Analytical methods:

The certified value for water-soluble arsenate extracted using water was determined by HPLC-ICP-MS under the following three analytical conditions:

Analytical condition 1: Ultrasonic extraction / ODS column

Analytical condition 2: Microwave-assisted extraction (60 °C) / ODS column

Analytical condition 3: Microwave-assisted extraction (60 °C) / Anion exchange column

Analysis

The certified values are the weighted means of results obtained using two or more analytical methods at NMIJ:

- (1) A single primary method and one or more reference methods
- (2) Three or more reference methods

The calibration solutions were prepared by a gravimetric method. The expanded uncertainty of the certified value is given by $U = k u_c$, where u_c is the combined standard uncertainty derived from the analytical results, between-method variance, sample inhomogeneity, and standard solution, and k is a coverage factor of 2, corresponding to a confidence interval of approximately 95%.

Metrological Traceability

For the trace elements, the certified values were determined by ID-ICP-MS carried out using Japan Calibration Service System (JCSS) standard solutions and are traceable to the International System of Units (SI). For arsenate, the certified value was determined by a method using NMIJ CRM 7912-a (arsenate solution) and is traceable to SI.

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 was prepared from *Hijiki* seaweed, which was powdered by freeze-pulverization after being freeze-dried. This CRM is in the form of a brown powder, filled in amber glass bottle (about 20 g each).

Homogeneity

The homogeneity of this CRM was determined by analyzing 10 bottles hierarchical-randomly selected from 400 bottles. The concentrations of trace elements were measured by ICP-MS or ICP-OES after microwave acid digestion. The concentration of arsenate was measured by HPLC-ICP-MS after ultrasonic extraction using water. The homogeneity is reflected in the uncertainty of the certified values.

Instructions for Storage

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

Instructions for Use

- 1) This CRM should be used up as soon as possible after opening to prevent contamination.
- 2) A dry mass correction is required when the CRM is analyzed. The correction factor is obtained by the following procedure.
 - (1) Approximately 1.0 g of the CRM is weighed in a weighing glass vessel, and is then heated at 85 °C for 12 h to 16 h.
 - (2) The combined mass of the CRM and vessel after cooling in a silica gel desiccator is measured.
 - (3) The difference in the CRM mass before and after drying is assumed to be the moisture content of the CRM.
 - (4) The sample used for correction should not be reused.The dry mass correction factor at the time of certification was about 5.7 %.
- 3) The following points should be adhered to when weighing the CRM.
 - (1) Weighing should not be performed under high humidity conditions.
 - (2) Weighing should be performed as quickly as possible.
 - (3) The CRM bottle should not be left open.
 - (4) Dry mass correction should be performed for every analysis.
- 4) From the viewpoint of homogeneity, more than 0.5 g of CRM should be used for each analysis.

Precautions for Handling

This CRM is for laboratory use only and is not edible. Care should be taken to prevent injuries when the bottle is opened, and a protective mask and gloves should be worn for safety when the CRM is used. All relevant laws regarding waste handling and management must be obeyed when the CRM is disposed. Refer to the safety data sheet (SDS) on this CRM before use.

Preparation

The *Hijiki* seaweed used for preparing this CRM was collected from the eastern coast of Japan. *Hijiki* was washed, freeze-dried, freeze-pulverized, sieved, and mixed for homogenization. The homogenized sample was packaged into amber glass bottles (about 20 g each) and was sterilized by ⁶⁰Co γ radiation (20 kGy). The bottles were sealed individually in aluminum-coated nylon packages. These preparation procedures were carried out by Japan Chemical Analysis Center (Japan), Funtai Giken Co., Ltd. (Japan), Radiation Application Development Association, Takasaki Establishment (Japan), and Asahi-rika Seisakusho Co., Ltd. (Japan).

Technical Information

The concentrations of mercury, expressed as mass fractions on a dry-mass basis, are provided as information values. The concentration of mercury was found to be 0.043 mg/kg as determined by isotope dilution–high-resolution ICP-MS after microwave acid digestion.

NMIJ Analysts

For this CRM, the technical manager is K. Chiba, the production manager is T. Narukawa, and the analysts are T. Narukawa, K. Inagaki, Y. Zhu, T. Kuroiwa, I. Narushima, and Y. Jimbo.

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.

Note

The certified values of the trace elements were validated by a coanalysis program performed by the Asian Collaboration on Reference Materials (ACRM) that has been jointly constructed by NMIJ (Japan), KRISS (Korea), and NIM (China) as part of the JST Strategic International Cooperation Program (SICP) 2004-2007.

April 1, 2015

Ryoji Chubachi
President

National Institute of Advanced Industrial Science and Technology

If you have any questions about this CRM, please contact:
National Institute of Advanced Industrial Science and Technology,
National Metrology Institute of Japan,
Center for Quality Management of Metrology, Reference Materials Office,
1-1-1 Umezono, Tsukuba, Ibaraki 305-8563, Japan
Phone: +81-29-861-4059; Fax: +81-29-861-4009, <https://unit.aist.go.jp/nmij/english/refmate/>

Revision history

April 1, 2015: "Metrology Management Center" was renamed to "Center for Quality Management of Metrology."