

National Institute of Advanced Industrial Science and Technology

## National Metrology Institute of Japan



## Reference Material Certificate

NMIJ CRM 7405-a  
No. 178Trace Elements and Arsenic Compounds in Seaweed (*Hijiki*)

This certified reference material (CRM) was produced in accordance with NMIJ's management system, and in compliance with JIS Q 0034 (ISO Guide 34). It is intended for use in evaluating or validating 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 following tables. They are expressed in mass fractions after correcting their dry mass. The drying method is described in this certificate. The quoted uncertainty 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 in the certified value is given by  $U = ku_c$ , 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

The certification of this CRM 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. The CRM is in the form of a dark green powder. It was filled in amber glass bottles (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.

### Precautions for Storage

This CRM should be stored in a clean place at room temperature (15 °C to 35 °C) and shielded from bright lights.

### Instructions for Use

- 1) Care should be taken when opening the bottle to prevent any contamination. Moreover, the CRM should be used up as soon as possible after opening.
- 2) 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) For maintaining homogeneity, more than 0.5 g of the CRM should be used in the analysis.

### **Instructions for Handling**

This CRM is for laboratory use only. It is not edible. Care should be taken to prevent any injuries during the opening of the bottle. A protective mask and safety gloves should be worn when handling the CRM. Lastly, laws regarding waste disposal should be followed when disposing of the CRM.

### **Preparation Method**

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  $^{60}\text{Co}$   $\gamma$  radiation (20 kGy). The bottles were sealed individually in aluminum-coated nylon packages.

### **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.

### **Collaborating Laboratory for Sample Preparation**

Japan Chemical Analysis Center, Japan

Funtai Giken Co., Ltd., Japan

Radiation Application Development Association, Takasaki Establishment, Japan

Asahi-rika Seisakusho Co., Ltd., Japan

### **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.

### **Technical Information**

Customer registration on the NMIJ Website (given below) will facilitate notification of any revision of the information given above. Technical reports regarding this CRM can be obtained from the contact details given below.

### **Additional Statement**

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.

**Reproduction of Certificate**

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

May 19, 2010

Tamotsu Nomakuchi

President

National Institute of Advanced Industrial Science and Technology

If you have any questions about this CRM, please contact:  
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Note: This certificate is a translation of the original Japanese certificate and is not an official document.