

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 based on NMIJ's quality system in compliance with JIS Q 0034 (ISO GUIDE 34), for use in evaluating or validating analytical methods and instruments used for the determination of the elements listed below and arsenate in *Hijiki* seaweed or similar matrices.

#### Certified Values

The certified values for 18 elements and water soluble arsenate extracted by water in this CRM are given in the following table. They are expressed in mass fractions after correcting dry mass. The drying instruction is described in this certificate. The expanded uncertainties were determined using coverage factor  $k = 2$ , corresponding to an estimated confidence interval of approximately 95 %.

Elements	Certified values, Mass fraction (g/kg)	Expanded uncertainty, Mass fraction (g/kg)	Analytical methods*
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

Elements	Certified values, Mass fraction (mg/kg)	Expanded uncertainty, Mass fraction (mg/kg)	Analytical methods*
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 atomic emission spectrometry (ICP-AES)

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- 5) Graphite furnace atomic absorption spectrometry
- 6) Flame atomic absorption spectrometry
- 7) Flame photometry

(Microwave acid digestion was performed for the 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 water soluble arsenate extracted by water was determined by HPLC-ICP-MS with 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

### Determination of Certified Values

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

- (1) Single primary method and one or more reference methods
- (2) Three or more reference methods.

All preparation was carried out by a gravimetric preparation method. The expanded uncertainty in the certified value is equal to  $U = k u_c$ , where  $u_c$  is the combined standard uncertainty derived from the analytical results, the between-method variance, the sample inhomogeneity and the standard solution, with coverage factor ( $k = 2$ ) corresponding to a 95 % confidence interval.

### Traceability

For the trace elements, the certified values were determined by ID-ICP-MS with JCSS (Japan Calibration Service System) standard solutions and are traceable to the International System of Units (SI). For the arsenate, the certified value was determined by a method with 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 is unopened and 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 freeze-dried. The CRM is in dark green powder form, which was bottled into an amber glass bottle (ca. 20 g each).

### Homogeneity

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

### Precautions for Storage

This CRM should be kept in a clean place at room temperature (15 °C—35 °C) and shielded from light.

### Precautions for Use

- 1) Be careful to contamination when opening, and it is desirable to use up as immediately as possible.
- 2) Dry mass correction is required when the CRM is analyzed. The correction factor was obtained by the following procedure.
  - ① ca. 1.0 g of the CRM is weighted into a weighting glass vessel. The CRM in the vessel is heated at 85 °C for 12-16

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hours.

- ② Weigh the CRM with the vessel after cooling in a silica gel desiccator.
- ③ The difference of the mass before and after drying is assumed moisture content.
- ④ Do not reuse the sample, which was used for the correction.

The dry mass correction factor at the time of the certification was ca. 5.7 %.

3) Be careful of the following points when the CRM is weighed.

- ① Do not weigh in a high humidity condition.
- ② Weighting have to be done as quick as possible.
- ③ Do not keep the bottle open.
- ④ Dry mass correction have to be done for every analysis.

4) From the homogeneity, the CRM should be used over 0.5 g for the analysis.

### **Precautions for Handling**

This CRM is for laboratory use only. This CRM is not edible, and do not eat. Be care to injuries when the bottle is opened. Use the protective mask and gloves for safety when the CRM is used. Obey the law about a waste when the CRM is disposed.

### **Preparation Method**

The *Hijiki* seaweed for this CRM was collected at the east coast of the Japan. The *Hijiki* was washed, freeze-dried, freeze-pulverized, sieved and mixed for homogenization. The homogenized sample was packaged into amber glass bottles (ca. 20 g each) and was sterilized by  $^{60}\text{Co}$   $\gamma$  radiation (20 kGy). The bottles were sealed individually in aluminum coated nylon packages.

### **References information**

The concentrations for the mercury, expressed as mass fractions on a dry-mass basis, are provided as information value. The concentration of mercury is 0.043 mg/kg that was 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**

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

### **Technical Information**

Customers will be notified in the case of important revision, such as a change in the certified values. Technical information about this CRM can be obtained from the contact shown below.

### **Additional Statement**

The certified values of the trace elements were validated in a co-analysis program performed by the “ACRM (Asian Collaboration on Reference Materials)” that is constructed with NMIJ (Japan), KRISS (Korea) and NIM (China) which is accomplishment 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.

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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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