National Institute of Advanced Industrial Science and Technology

National Metrology Institute of Japan

Reference Material Certificate

NMIJ CRM 7502-a
No. +++

Trace Elements in White Rice Flour (Cd Level II)

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 analytical methods and instruments during the analysis of arsenic compounds and trace elements in rice and other grains.

Certified Values

The certified values for 17 trace elements, inorganic arsenic compounds (total amount of arsenite and arsenate), and dimethylarsinic acid in this CRM are given in the tables below. The values are expressed in mass fractions based on dry mass. The drying procedure is given 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 methods (see below)
Cr	0.075	0.013	2, 3
Mn	11.2	0.4	3, 5, 6
Fe	4.48	0.20	1, 3, 5, 6
Ni	0.390	0.022	1, 3
Cu	3.02	0.11	1, 3, 6
Zn	26.0	0.9	1, 3, 6
As	0.109	0.005	3, 4, 6
Rb	1.77	0.07	1, 3
Sr	0.068	0.003	1, 3
Mo	0.79	0.03	1, 3
Cd	0.548	0.020	1, 3, 6
Pb	0.0043	0.0006	2, 3
Na	5.8	0.8	5, 7, 8
Mg	560	21	3, 5, 7
P	1800	90	3, 4, 5
K	1430	50	5, 7, 8
Ca	60	3	3, 5, 8

Analytical methods:

1) Isotope dilution-inductively coupled plasma mass spectrometry (ID-ICP-MS)
2) ID-high-resolution ICP-MS
3) ICP-MS
4) High-resolution ICP-MS
5) ICP-optical emission spectrometry (ICP-OES)
6) Graphite furnace atomization atomic absorption spectrometry (GFAAS)
7) Flame-AAS

8) Flame photometry

(Microwave acid digestion or dry-ashing was performed for a sample pretreatment)

Compound	CAS No.	Certified value, Mass fraction (mg/kg)	Expanded uncertainty Mass fraction (mg/kg)
Inorganic arsenic compounds (arsenite + arsenate) (as As)	1327-53-3 (arsenite) 7778-39-4 (arsenate)	0.098	0.006
Dimethylarsinic acid (as As)	75-60-5	0.0129	0.0010

Analytical method: Heat extraction/high-performance liquid chromatography-inductively coupled plasma-mass spectrometry (HPLC-ICP-MS)

9. [Extraction method] Dry block bath/[Extract] 0.15 mol L^{-1} HNO_3/[Extraction temperature] 100 °C/[HPLC column] CAPCELL PAK C18MG/[Measurement] High-performance liquid chromatography-inductively coupled plasma mass spectrometry
10. [Extraction method] Dry block bath/[Extract] 0.15 mol L^{-1} HNO_3/[Extraction temperature] 100 °C/[HPLC column] Shim-pack VP-C8/[Measurement] High-performance liquid chromatography-inductively coupled plasma mass spectrometry
11. [Extraction method] Dry block bath/[Extract] 0.0015 mol L^{-1} HNO_3/[Extraction temperature] 100 °C/[HPLC column] CAPCELL PAK C18MG/[Measurement] High-performance liquid chromatography-inductively coupled plasma mass spectrometry

Analysis

The certified values of this CRM are the weighted means of the results from two or more analytical methods conducted at NMIJ. The quantitative analysis of elements was made by the aforementioned analytical methods [1)–8)], and combinations of these are based on (1) a single primary method (ID-ICP-MS) with one or more reference methods or (2) three or more reference methods. The quantitative analysis of arsenic compounds was made after extraction with weak nitric acid (HNO_3) by HPLC-ICP-MS. Three different analytical methods were used with combinations of levels of HNO_3 concentrations and types of reverse-phase HPLC columns.

The expanded uncertainty of 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 dry mass correction, (d) the concentration of a standard solution, and (e) the sample homogeneity.

Metrological Traceability

The certified values were determined by isotope dilution mass spectrometry or other accurate methods with JCSS (Japan Calibration Service System) standard solutions and NMIJ CRMs (NMIJ CRM 7912-a arsenate [As(V)] solution and NMIJ CRM 7913-a dimethylarsinic acid solution), and all are traceable to the International System of Units (SI). All sample preparation was carried out by the gravimetric method using a balance calibrated by JCSS.

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

The certificate is valid from the data of shipment to March 31, 2023, provided that the material remains unopened and is stored in accordance with the instructions given in this certificate.

Sample Form

This CRM is prepared from rice that was powdered by freeze-pulverization after polishing. This CRM is in the form of white flour and kept in an amber glass bottle (20 g each).

Homogeneity

The homogeneity of this CRM was determined by analyzing 10 bottles from a hierarchically random sampling of 650 bottles. Each trace element was determined by ICP-MS or ICP-OES after microwave acid digestion. Each arsenic compound was determined by HPLC-ICP-MS after extraction with weak nitric acid. The inhomogeneity of the analytes, which was evaluated by ANOVA, was not significant and is reflected in the uncertainty of the certified value. This material is homogeneous within the range of the uncertainty of the certified value.

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) Take care to prevent contamination of the CRM when opening the bottle and use up the CRM immediately.
2) Dry mass correction is required when the CRM is analyzed. The correction factor is obtained by the following procedure:
    (1) Weigh *ca.* 0.5 g of the CRM into a weighing glass vessel and then, heat it at 95 °C for 12 to 16 h.
    (2) Weigh the CRM with the vessel after cooling in a desiccator.

The difference in the mass before and after drying is assumed to be the moisture content. The dry mass correction factor at the time of the certification was *ca.* 10.5 %. Do not use the sample that was used for the dry mass correction for analysis.

3) Note the following points when the CRM is weighed:
    (1) Do not weigh in a high humidity conditions (over 60%).
    (2) Weighing must be done as quickly as possible.
    (3) Do not keep the bottle open.
    (4) Dry mass correction must to be done for every analysis.
4) From the homogeneity, more than 0.5 g for the analysis of trace elements and arsenic compounds.

Precautions for Handling

This CRM is for laboratory use only. Take care to prevent injuries when the bottle is opened. Use a protective mask and gloves for safety when this CRM is used. All relevant laws regarding waste handling and management must be obeyed when disposing of this CRM. Refer to the safety data sheet (SDS) on this CRM before use.

Preparation

This CRM was prepared from polished rice. The rice was powdered by freeze-pulverization. The powder was placed into amber glass bottles (20 g each) by using a split method and sterilized with γ-ray irradiation (^{60}Co, 20 kGy). The bottles were sealed individually in polypropylene packages and stored at room temperature. The preparation of the candidate material and the γ-ray irradiation were performed by KANSO Technos and Radiation Application Development Association, respectively.

NMIJ Analysts

The technical manager is CHIBA K., the production manager is INAGAKI K., and the analysts are INAGAKI K., NARUKAWA T., ZHU Y., JIMBO Y., and NARUSHIMA I.

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

This CRM was developed based upon completion of a project supported by the SME Intellectual Foundation Construction Project of Ministry of Economy 2006-2007, Trade and Industry, Japan.

April 1, 2020

ISHIMURA Kazuhiko
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
January 7, 2015: The certified values of inorganic arsenic compounds (arsenite + arsenate) and dimethylarsinic acid were added.
January 7, 2015: 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.”
March 31, 2022: The certification items were reviewed.