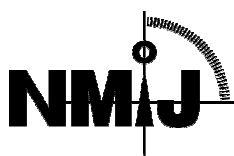


National Institute of Advanced Industrial Science and Technology

National Metrology Institute of Japan



Reference Material Certificate

NMIJ CRM 7503 - a
No. +++



Arsenic Compounds and Trace Elements in White Rice Flour

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 controlling the precision of analysis or to confirm the validity of analytical methods or instruments during the analysis of arsenic compounds and/or trace element in rice flour.

Certified Values

The certified values for three arsenic compounds and six trace elements 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 %.

| Compounds | Certified values, Mass fraction (mg/kg, as As) | Expanded uncertainty (mg/kg, as As) |
|-----------------------------|---|--|
| Arsenite (As(III)) | 0.0711 | 0.0029 |
| Arsenate (As(V)) | 0.0130 | 0.0009 |
| Dimethylarsinic acid (DMAA) | 0.0133 | 0.0009 |

Analytical methods;

Microwave assisted extraction (solvent: water) /

high performance liquid chromatography – inductively coupled plasma mass spectrometry (HPLC-ICP-MS)

Analytical condition 1: [Extraction Temp.]: 60°C [HPLC column]: L-column (CERI)

Analytical condition 2: [Extraction Temp.]: 80°C / [HPLC column]: L-column (CERI)

Analytical condition 3: [Extraction Temp.]: 80°C / [HPLC column]: Develosil C30-UG-5 (Nomura Chemical)

Analytical condition 4: [Extraction Temp.]: 80°C / [HPLC column]: IC PRP-X100 (Hamilton)

| Element | Certified values Mass fraction (mg/kg) | Expanded uncertainty (mg/kg) | Analytical methods* |
|---------|---|---------------------------------|------------------------|
| Mn | 9.2 | 0.4 | 2, 4, 5 |
| Fe | 5.42 | 0.21 | 1, 2, 4 |
| Cu | 1.88 | 0.07 | 1, 2 |
| Zn | 20.7 | 0.9 | 1, 2 |
| As | 0.098 | 0.007 | 2, 3, 5 |
| Cd | 0.194 | 0.007 | 1, 2 |

Analytical methods;

- 1) Isotope dilution -ICP-MS
- 2) ICP-MS
- 3) High resolution ICP-MS
- 4) ICP optical emission spectrometry (ICP-OES)
- 5) Graphite furnace atomization atomic absorption spectrometry (GFAAS)
(Microwave acid digestion or dry-ashing is performed for a sample pretreatment)

[This document is just explanation translated from the original Japanese certificate and some information is omitted from it.]

Determination of Certified Values

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

- (1) ID-ICP-MS(a primary method) and one or more reference methods
- (2) More than three reference methods.

The uncertainty of the certified value was obtained by combination of the uncertainty of (a) the analytical method, (b) bias among the analytical methods, (c) inhomogeneity, and (d) dry mass correction.

Traceability

For the arsenic compounds, the certified values were determined by a method with JCSS standard solution (As(III)), NMIJ CRM 7912-a (As(V)), and NMIJ CRM 7913-a (dimethylarsinic acid) and are traceable to the International System of Units (SI). For the trace elements, the certified values were determined by ID-ICP-MS with JCSS standard solutions and are traceable to the SI.

Global mutual recognition

This CRM is included in the calibration and measurement capability as stated in the database of the International Bureau of Weights and Measures (BIPM) based on a Global Mutual Recognition Arrangement (CIPM-MRA), under the authority given to it in the Meter Convention.

Expiration of Certification

The certification of this CRM is valid until March 31, 2024, provided that the CRM is unopened and stored in accordance with the instructions given in this certificate.

Sample Form

This CRM was prepared from rice. The rice was powdered by freeze-pulverization after polishing. The form of this CRM is white flour, which was placed into an amber glass bottle (20 g each).

Homogeneity

The homogeneity of the CRM was determined by analyzing 10 bottles hierarchical-randomly selected from 650 bottles. The arsenic compounds were determined by HPLC-ICP-MS after microwave assisted extraction. The trace elements were determined by ICP-MS after microwave acid digestion. The homogeneity is reflected in the uncertainty of the certified values.

Precaution for Storage

This CRM should be kept in a clean place at room temperature (under 28 °C) and shielded from light.

Precaution for Use

- (1) Be careful to contamination when the bottle is opened, and use it up immediately.
- (2) Dry mass correction is required when the CRM is analyzed. The correction factor was obtained by the following procedure.
 - ① ca. 0.5 g of the CRM is weighted into a weighting glass vessel. The CRM in the vessel is heated at 95 °C for 12-16 hours.
 - ② Weigh the CRM with the vessel after cooling in desiccator.
 - ③ Do not use the sample, which used for the correction, for analysisThe dry mass correction factor at the time of the certification was ca. 6.7 %.
- (3) Be careful to the following points when the CRM is weighed.
 - ① Do not weigh in a high humidity condition (over 60 %).
 - 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 1g for the analysis of the arsenic compounds and over 0.5 g for the analysis of the trace elements.

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Precaution for Handling

This CRM is for laboratory use only. This CRM is not edible, and do not eat. Be careful 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 CRM was prepared from rice polished. The rice was powdered by freeze-pulverization. The powder was placed into amber glass bottles (20 g each) by using a split method and was sterilized by ^{60}Co γ radiation (20 kGy). The bottles were sealed individually in polypropylene packages. They are stored at room temperature.

NMIJ Analyst

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

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

This CRM was developed based on the accomplishment of the project supported by the SME intellectual foundation construction project of Ministry of Economy 2006-2007, Trade and Industry Japan.

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), KRIS (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.

August 4, 2009

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