

National Institute of Advanced Industrial Science and Technology

National Metrology Institute of Japan



Reference Material Certificate

NMIJ CRM 7531-a

No. +++



Cadmium in Brown Rice Flour

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 cadmium and trace elements in rice and other grains.

Certified Values

The certified values of this CRM are given in the table below. The value is expressed as a mass fraction, based on a dry mass (the drying procedure is given in this certificate). The uncertainty of the certified value is the expanded uncertainty obtained by multiplying the combined standard uncertainty by a coverage factor (k) of 2, and it is the half-width of an interval estimated to have a level of confidence of approximately 95 %.

Element	Certified value Mass fraction (mg/kg)	Expanded uncertainty Mass fraction (mg/kg)	Analytical methods (see below)
Mn	27.6	0.7	2, 4, 5
Fe	11.66	0.32	1, 2, 4
Cu	4.34	0.13	1, 2, 4
Zn	31.8	0.7	1, 2, 4
As	0.280	0.009	2, 3, 5
Cd	0.308	0.007	1, 2, 4

Analytical methods

- 1) Isotope dilution-inductively coupled plasma mass spectrometry (ID-ICP-MS)
- 2) Inductively coupled plasma mass spectrometry (ICP-MS)
- 3) High-resolution inductively coupled plasma mass spectrometry
- 4) Inductively coupled plasma optical emission spectrometry
- 5) Graphite furnace atomic absorption spectrometry

The sample digestion method for 1) and 3) was microwave acid digestion with nitric acid, hydrofluoric acid, and perchloric acid. The digestion method for all others was microwave acid digestion with nitric acid, hydrofluoric acid, and hydrogen peroxide.

Analysis

The certified value of this CRM was weighted mean of the results of the following analytical methods:

- (1) Single primary method (ID-ICP-MS) and one or more reference methods
- (2) Three or more reference methods

The expanded uncertainty in 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 the standard solution, and (e) the sample homogeneity.

Metrological Traceability

The certified value was determined by the primary method (ID-ICP-MS) or other accurate reference methods, with JCSS (Japan Calibration Service System) standard solutions. All sample preparation was carried out by a gravimetric method, using a balance calibrated by JCSS. The certified value, therefore, is traceable to the International System of Units (SI).

Mutual Recognition Arrangement under Metre Convention

The certified values of this CRM are recognized for international equivalence based on the Mutual Recognition Arrangement under the Metre Convention (CIPM MRA). The calibration measurement capabilities (CMC) of NMIJ related to this CRM are registered in the Key Comparison Database (KCDB) (see <https://www.bipm.org/kcdb/>) of the International Bureau of Weights and Measures (BIPM).

Expiration of Certification

This certificate is valid for one year from the date of shipment, provided that this CRM is stored in accordance with the instructions given in this certificate.

Description of the Material

This CRM is in the form of light brown flour of domestic rice in Japan and ca. 20 g in net volume is kept in an amber glass bottle.

Homogeneity

The homogeneity of this CRM was determined by analyzing 10 bottles from a hierarchically random sampling of 640 bottles. Each element was determined by ICP-MS after microwave acid digestion with nitric acid, hydrofluoric acid, and hydrogen peroxide. The inhomogeneity of each element, which was evaluated by analysis of variance (ANOVA), is not significant and is reflected in the uncertainty of the certified value. This material is homogeneous within the uncertainty of the certified value.

Instructions for Storage

This CRM should be stored at temperatures of 5 °C to 30 °C in a clean place and protected from light.

Instructions for Use

- 1) This CRM should be opened and used up as soon as possible after opening to prevent contamination. When the bottle is stored after opening, it should be sealed with tape and kept in a desiccator with silica gel to limit its absorption of moisture as much as possible.
- 2) Dry mass correction is required when the CRM is analyzed, as each certified value is expressed as a mass fraction, based on a dry mass. The correction factor should be obtained by the procedure shown below. Do not use the sample that is used for the correction for analysis.
 - (1) Take ca. 0.5 g of the CRM in a weighing glass vessel.
 - (2) Dry the CRM in the vessel at 95 °C for 12 h to 16 h in a drying oven.
 - (3) Weigh the CRM with the vessel after cooling in a desiccator with silica gel for 30 min.
 - (4) The difference in the masses before and after drying is assumed to be the moisture content. The dry mass correction factor at the time of the certification was ca. 5.1 % (mass fraction).
- 3) Care should be taken to address the following points when the CRM is weighed.
 - (1) Do not weigh it in conditions of high humidity (over 60 %).
 - (2) Weighing needs to be performed as quickly as possible.
 - (3) Do not leave the bottle open when it is not in use, in order to minimize the time the CRM is exposed to the atmosphere.
 - (4) Weighing for dry mass correction has to be done in parallel with weighing for analysis.
- 4) Considering the homogeneity, a minimum sample size of 0.5 g should be used to ensure valid results.

Precautions for Handling

This CRM is for laboratory use only, and is not edible. Wear a protective mask and gloves during handling. Obey relevant law regarding waste disposal. Refer to the safety data sheet (SDS) on this CRM before use.

Preparation

Approximately 30 kg of fresh brown rice, for which the average Cd concentration and coefficient of variation (homogeneity) were confirmed to be in a range of 0.20 mg/kg to 0.35 mg/kg and $\leq 15\%$ ($n = 10$), respectively, was obtained from a domestic market and used for preparation of the CRM. The fresh brown rice was dried at 60 °C for 8 h, and then freeze pulverized. The brown rice flour was again dried at 60 °C for 8 h, and then placed into amber glass bottles (about 20 g in each) using a splitting method. The bottles were individually vacuum sealed into seal bags (Lamizip Aluminum). Finally, the candidate CRM was sterilized with ^{60}Co γ -ray irradiation (about 20 kGy). The preparation of the candidate CRM and the γ -ray irradiation were performed by KANSO Technos and Radiation Application Development Association, respectively.

NMIJ Analysts

The technical manager for this CRM is HIOKI A., the production manager is INAGAKI K., and the analysts are MIYASHITA S., INAGAKI K., NARUKAWA T., ZHU Y., KOGUCHI M., and KUDO I.

Information

If substantive technical changes occur that affect the certification before the expiration of this certificate, NMIJ will notify the registered customers. 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.

April 1, 2020

ISHIMURA Kazuhiko
President

National Institute of Advanced Industrial Science and Technology

If you have any questions about this CRM, please contact:
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Revision history

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

February 13, 2018: The description in "Expiration of Certification" was changed to "one year from the date of shipment."