

National Institute of Advanced Industrial Science and Technology

## National Metrology Institute of Japan



## Reference Material Certificate

NMIJ CRM 7512-a

No. +++



## Trace Elements in Milk Powder

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 the calibration of instruments, and validation of analytical methods and instruments used for the quantification of trace elements in milk powder or similar matrices.

## Certified Values

The certified values of this CRM are given in the table below. The uncertainty of each 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 (g/kg)	Expanded uncertainty, Mass fraction (g/kg)	Analytical method*
Ca	8.65	0.38	2, 3, 4, 6, 7
Fe	0.104	0.007	1, 2, 4
K	8.41	0.33	2, 4, 6, 7
Mg	0.819	0.024	2, 3, 4, 6
Na	1.87	0.09	2, 3, 4, 6, 7
P	5.62	0.23	2, 3, 4
Element	Certified value, Mass fraction (mg/kg)	Expanded uncertainty, Mass fraction (mg/kg)	Analytical method*
Ba	0.449	0.013	1, 2, 3
Cu	4.66	0.23	1, 2, 3, 5
Mn	0.931	0.032	2, 3, 5
Mo	0.223	0.012	1, 2, 3
Rb	8.93	0.31	1, 2, 3
Sr	5.88	0.20	1, 2, 3
Zn	41.3	1.4	1, 2, 3, 4

\*Analytical methods (microwave assisted acid digestion was performed as the sample pretreatment prior to the analysis):

- 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
- 6) Flame atomic absorption spectrometry
- 7) Flame atomic emission spectrometry

The sample digestion method for 1) to 3) was a microwave digestion with nitric acid, hydrogen peroxide and hydrofluoric acid.

The digestion method for the others was a microwave digestion with nitric acid, perchloric acid and hydrofluoric acid.

## Analysis

These certified values of this CRM were the weighted means of the results from two or more analytical methods conducted at

NMIJ. The quantitative analyses of elements were made by the aforementioned analytical methods of 1) to 7), 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 expanded uncertainty in each certified value is equal to  $U = k \cdot u_c$ , where  $u_c$  is the combined standard uncertainty derived from: (a) the analytical results, (b) the method-to-method variance, (c) the sample homogeneity, (d) the dry mass correction, and (e) the concentration of a standard solution.

### **Metrological Traceability**

Each certified value was determined by multiple methods with standard solutions guaranteed by JCSS (Japanese Calibration Service System) that traceable to the International System of Units (SI). All the working standard and sample solutions were prepared by a gravimetric method, using a balance calibrated by JCSS. The certified values, therefore, are 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 (CMCs) 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 was prepared from infant formula milk powder for 6-month old children. The material was powdered by using a laboratory mill. The CRM is in milky white powder form, which was bottled into amber glass bottles (*ca.* 40 g each).

### **Instructions for Storage**

This CRM should be stored at temperatures of 5 °C to 35 °C and protected from light.

### **Instructions for Use**

- 1) Attention should be paid to avoid contamination when the bottle is opened. It is also desirable to use up as soon as possible.
- 2) From the viewpoint of homogeneity, it is desirable to shake the bottle at least 5-times before taking the sample. Sample amount over 0.3 g is recommended for quantification.
- 3) Dry mass correction is required when the CRM is analyzed. The correction factor should be obtained by the following procedure:
  - ① Approximately 0.3 g of the CRM is quickly weighed into a weighing glass vessel. The CRM in the vessel is heated at 65 °C for 15 h to 25 h.
  - ② Weigh the CRM with a vessel after cooling for 30 min in a silica gel desiccator.
  - ③ The difference of the masses before and after drying is assumed as moisture content.
  - ④ Do not use the sample in dry mass correction for digestion or analysis.The dry mass correction factor at the time of the certification was *ca.* 2.2 % (mass fraction).
- 4) Be careful of the following points when the CRM is weighed.
  - ① Do not weigh in a high humidity condition.
  - ② Weighing has to be done as quickly as possible.
  - ③ Do not keep the bottle open.

### **Precautions for Handling**

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This CRM is for laboratory use only. This CRM is not edible. 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

The infant formula milk powder was purchased from a bulk batch of a commercial company. The milk powder was further powdered with a laboratory mill, sieved and mixed for homogenization. The obtained milk powder was placed into amber glass bottles (*ca.* 40 g each) by using a split method and was sterilized with  $\gamma$ -ray irradiation ( $^{60}\text{Co}$ , 25 kGy). The bottles were labeled and sealed individually in polypropylene packages and have been stored at room temperature. The preparation procedures were carried out by the Korea Research Institute of Standards and Science with exception of labeling and sealing by NMIJ.

### Technical Information

The concentrations of As, Cd, Cr, Pb, and Y were summarized in the follow table as information values. The concentrations of these elements were expressed as mean values of the analysis after dry mass correction. Microwave assisted acid digestion was carried out to obtain the reference information.

Element	Reference information, Mass fraction( $\mu\text{g/kg}$ )	Analytical method (refer to those for certified values)
As	2.1	3
Cd	0.2	3
Cr	1.3	3
Pb	0.3	3
Y	64	2, 3

### NMIJ Analysts

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

### 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.

### Note

The certified values of the trace elements were additionally validated in a co-analysis program “Trace Elements in Milk Powder” performed by the “ACRM (Asian Collaboration on Reference Materials)” that is constructed with NMIJ (Japan), KRISS (Korea) and NIM (China). The candidate reference materials for the present CRM was provided by KRISS.

April 1, 2020

Date of Shipment: Xxxxx xx, 20xx

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Revision history

April 1, 2015: "Metrology Management Center" was renamed to "Center for Quality Management of Metrology."  
March 25, 2021: The description in "Expiration of Certification" was changed to "one year from the date of shipment".