

National Institute of Advanced Industrial Science and Technology

National Metrology Institute of Japan



Reference Material Certificate

NMIJ CRM 7511-a

No. +++



Trace Elements in Soybean

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 confirming the validity of analytical methods and instruments during the quantification of trace elements in soybean and similar matrix samples.

Certified Values

The certified values for 8 elements in this CRM are given in the table below. They are expressed in mass fractions after correcting dry mass. The drying instruction is described 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 (g/kg) | Expanded uncertainty Mass fraction (g/kg) | Analytical method See below |
|---------|---|--|--------------------------------|
| Ca | 2.76 | 0.08 | 1, 5, 7, 8 |
| K | 18.6 | 0.8 | 5, 7, 8 |
| Mg | 2.74 | 0.10 | 1, 5, 7 |
| P | 7.4 | 0.5 | 1, 3, 5 |

| Element | Certified value Mass fraction (mg/kg) | Expanded uncertainty Mass fraction (mg/kg) | Analytical method See below |
|---------|--|---|--------------------------------|
| Cd | 0.147 | 0.009 | 1, 2, 4, 5, 6 |
| Cu | 11.8 | 0.5 | 1, 2, 5, 6 |
| Fe | 69.1 | 2.4 | 1, 2, 5, 6 |
| Zn | 57.8 | 1.7 | 1, 2, 5 |

*Analytical methods;

- 1) Inductively coupled plasma mass spectrometry with quadrupole system (ICP-MS with quadrupole system)
- 2) Isotope dilution ICP-MS with quadrupole system (ID-ICP-MS with quadrupole system)
- 3) ICP-MS with double focusing system
- 4) ID-ICP-MS with double focusing system
- 5) Inductively coupled plasma optical emission spectrometry (ICP-OES)
- 6) Graphite furnace atomic absorption spectrometry
- 7) Flame atomic absorption spectrometry
- 8) Flame photometry

(Microwave acid digestion was performed for the sample pretreatment)

Analysis

The certified values of this CRM are the weighted means of the results from two or more analytical methods mentioned above. Combination of these methods are based on:

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

(2) Three or more reference methods.

All preparation was carried out by the gravimetric preparation method. The expanded uncertainty (U) in each certified value is equal to ku_c , where u_c is the combined standard uncertainty derived from : (a) the measurements, (b) the between-method variance, (c) the dry mass correction factor, (d) the concentration of a standard solution, (e) the between-preparation variance and (f) the sample inhomogeneity, and k ($=2$) is the coverage factor corresponding to the 95 % confidence level.

Metrological Traceability

The certified values were determined by the ID-ICP-MS with quadrupole system, or other accurate methods, with JCSS (Japan Calibration Service System) standard solutions and all are traceable to the International System of Units (SI). All the working standards and sample solutions were prepared 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 <https://www.bipm.org/kcdb/>).

Expiration of Certification

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

Sample Form

This CRM is in the form of a brown white powder which was prepared from soybean. This CRM of ca. 30 g in net volume is kept in an amber glass bottle.

Homogeneity

The homogeneity of this CRM was determined by analyzing 10 bottles hierarchical-randomly sampled from 300 bottles. Each trace element was determined by ICP-MS with quadrupole system or ICP-OES after microwave acid digestion. 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 uncertainty of the certified values.

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) Before opening the bottle, shake gently at room temperature. Be careful of contamination when opening the bottle, then this CRM should be used up as soon as possible. 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) A 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 following procedure. Do not use the sample that is used for the correction for analysis.
 - ① Take ca. 1.0 g of the CRM into a weighing glass vessel. Dry the CRM in the vessel at 85 °C for 12 h to 16 h.
 - ② Weigh the CRM with the vessel after cooling in a silica gel desiccator with silica gel for 30 min.
 - ③ 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 certification was ca. 3.5 % (mass fraction).
- (3) Care should be taken to address the following points when the CRM is weighed, since this CRM is highly hygroscopic.
 - 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 the dry mass correction has to be carried out in parallel with weighing for analysis.

(4) From the viewpoint of homogeneity, more than 0.5 g of CRM should be used for each analysis.

Precautions for Handling

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

Preparation

The CRM was produced by Korea Research Institute of Standards and Science (KRISS, Korea) in a co-analysis program performed by the “ACRM (Asian Collaboration on Reference Materials).” The soybean was washed, freeze-dried, freeze-pulverized, sieved and mixed for homogenization. The homogenized sample was packaged into amber glass bottles (ca. 30 g each) and was sterilized by ^{60}Co γ radiation (20 kGy). The preparations of the candidate material were performed by KRISS. The bottles were individually vacuum sealed into nylon packages at NMIJ; then, all processes including the homogeneity test and the assigning the property values were carried out by NMIJ.

NMIJ Analysts

The technical manager is HIOKI A., the production manager is NARUKAWA T., and the analysts are NARUKAWA T., ZHU Y., INAGAKI K., MIYASHITA S., 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 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.

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."

September 17, 2020: The description in "Expiration of Certification" was changed to "one year from the date of shipment."

The description on "Mutual Recognition Arrangement under Meter Convention" was added.

Sample