

National Institute of Advanced Industrial Science and Technology

National Metrology Institute of Japan



Reference Material Certificate

NMIJ CRM 7505-a
No. +++

Trace Elements in Tea Leaf 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 use in controlling the precision of analyses and validating analytical methods and instruments used in the analysis of trace elements in tree leaves and similar matrices.

Certified Values

The certified values for 18 elements in this CRM are given in the table below. They are expressed in mass fractions corrected for dry mass. The drying instructions are 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 %.

Elements	Certified values, Mass fraction (%)	Expanded uncertainty, Mass fraction (%)	Analytical methods*
Ca	0.450	0.015	2, 3, 4, 6
K	1.59	0.05	2, 3, 4, 6
Mg	0.301	0.009	2, 3, 4, 6
P	0.339	0.011	2, 3, 4

Elements	Certified values, Mass fraction (mg/kg)	Expanded uncertainty, Mass fraction (mg/kg)	Analytical methods*
Al	709	26	2, 3, 4, 5
B	19.7	0.9	1, 2, 3
Ba	20.4	0.7	1, 2, 3, 4
Cd	0.0139	0.0012	1, 2, 3
Cu	19.2	0.8	1, 2, 3, 4, 5
Fe	82.1	2.8	1, 2, 3
Li	0.57	0.04	1, 2, 3
Mn	760	26	2, 3, 4, 5
Na	7.2	0.5	2, 3, 4, 6
Ni	5.5	0.3	1, 2, 3, 4
Pb	0.094	0.006	1, 2, 3
Rb	7.3	0.3	1, 2, 3
Sr	9.0	0.3	1, 2, 3, 4
Zn	22.7	0.8	1, 2, 3

*Analytical methods;

- 1) Isotope dilution-inductively coupled plasma mass spectrometry (ID-ICP-MS)
- 2) Inductively coupled plasma mass spectrometry (ICP-MS)
- 3) High resolution ICP-MS

- 4) Inductively coupled plasma atomic emission spectrometry (ICP-AES)
 - 5) Graphite furnace atomic absorption spectrometry
 - 6) Flame atomic absorption spectrometry
- (The microwave assisted acid digestion was employed for the sample pretreatment)

Analysis

The certified values of this CRM are the weighted means of results obtained from two or more analytical methods conducted at NMIJ: (1) ID-ICP-MS (primary method) and one or more reference methods and (2) Three or more reference methods. These analyses were conducted at NMIJ. The gravimetric preparation method was employed for all preparations. 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 sample homogeneity, (d) the dry mass correction, and (e) the concentration of the standard solution.

Metrological Traceability

The certified values of this CRM are determined by the isotope dilution mass spectrometry or other accurate methods, with the JCSS (Japan Calibration Service System) standard solutions, all of which are traceable to the International System of Units (SI). All the working standards and sample solutions are prepared by the gravimetric method, using a JCSS-calibrated balance.

Indicative Value

The concentration of Co in this CRM is given in the table below as an indicative value. It is expressed in mass fractions corrected for dry mass. The uncertainty of this indicative 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 %. The sample pretreatment, dry mass correction, homogeneity evaluation, and uncertainty evaluation were carried out in the same way as those for the certified values.

Element	Indicative value, Mass fraction (mg/kg)	Expanded uncertainty, Mass fraction (mg/kg)	Analytical methods (refer to those for certified values)
Co	0.257	0.012	2, 3

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 certification of this CRM is valid until March 31, 2030, 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 dark brown powder which was prepared from fresh tea leaves. This CRM of 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 selected with the stratified random sampling method from the total of 732 bottles. Each element was determined by the ICP-MS or the ICP-AES after being treated by the microwave assisted acid digestion. The inhomogeneity of the analytes, which was evaluated by the ANOVA, is not significant and it has been incorporated into 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) Once a bottle is opened, this CRM should be used up as soon as possible to prevent contamination.
- 2) To ensure the homogeneity, it is desirable to shake a bottle at least five times before taking the sample. More than 0.5 g of the material should be used for each analysis.
- 3) 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 procedures:
 - (1) Weigh approximately 0.5 g of the CRM promptly with a glass weighing vessel. Dry the CRM in the glass weighing vessel at 85 °C for 15-25 hours in a drying oven.
 - (2) Cool the CRM in a desiccator with silica gel for 30 minutes, and promptly weigh the CRM with the glass weighing vessel.
 - (3) The difference in the masses before and after the drying process is assumed to be the moisture content.
 - (4) The sample that is used for the correction must not be used for analysis.
The dry mass correction factor at the time of the certification was ca. 7.4 % (mass fraction).
- 4) Care should be taken to address the following points when weighing the CRM.
 - (1) Do not weigh this CRM under the conditions of high humidity.
 - (2) Perform weighing as promptly as possible.
 - (3) Do not leave a bottle of this CRM open when it is not in use, in order to minimize the time the CRM is exposed to the atmosphere.
 - (4) Carry out the weighing for the dry mass correction in parallel with the weighing for analysis.

Precautions for Handling

This CRM is for laboratory use only, and is not edible. Care should be taken to prevent injuries when opening a bottle, and a protective mask and gloves should be worn for safety when the CRM is used. This CRM must be disposed of in accordance with all relevant laws regarding waste handling and management. Refer to the safety data sheet (SDS) on this CRM before use.

Preparation

The tea leaves for this CRM were harvested from a tea farm in Shizuoka Prefecture, Japan. The tea leaves were washed, dried, freeze-pulverized, sieved and mixed for homogenization. The thus obtained tea leaf powder was packaged in amber glass bottles (20 g each) by using the split method and was sterilized with γ -ray irradiation (^{60}Co , 20 kGy). Each of the bottles was sealed in polypropylene package and stored at room temperature.

Technical Information

The concentrations of Ti, V, Cr, Y, and lanthanides were summarized in the table below as information. The concentrations of these elements were expressed as mean values of the analysis corrected for dry mass. The microwave assisted acid digestion was carried out to obtain this information.

Elements	Concentration, Mass fraction (mg/kg)	Analytical methods (refer to those for certified values)
Ti	0.6	1, 2, 3
V	0.07	2, 3
Cr	0.15	1
Y	0.10	2, 3, 4
La	0.10	3
Ce	0.12	3
Pr	0.028	3

Nd	0.10	3
Sm	0.025	3
Eu	0.0057	3
Gd	0.028	3
Tb	0.0040	3
Dy	0.023	3
Ho	0.0044	3
Er	0.012	3
Tm	0.0017	3
Yb	0.010	3
Lu	0.0014	3

NMIJ Analysts

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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

The certified values of the trace elements were validated in the co-analysis program “Trace Elements in Tea Leaf” performed by the “ACRM (Asian Collaboration on Reference Materials)” that is composed of NMIJ (Japan), KRISS (Korea) and NIM (China).

April 1, 2020

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President

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Revision history

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

February 21, 2017: The description of “Mutual Recognition Arrangement under Meter Convention” was added.