

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) was produced based on NMIJ's quality system in compliance with ISO GUIDE 34:2000 and ISO/IEC 17025:2005 for quality control, evaluation, or validation of analytical methods or analytical instruments in determination of trace elements in tree leaves or similar matrices.

Certified Values

The certified values for 18 elements in this CRM are given in the following table. They are expressed in mass fractions after dry mass correction. The drying instruction is described in this certificate. The expanded uncertainties were determined using a coverage factor of $k = 2$, which is corresponding to an estimated confidence interval 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

(Microwave assisted acid digestion was performed as the sample pretreatment)

Analytical Methods

The certified values are the weighted means of results from two or more analytical methods carried out at NMII:

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

All preparation was carried out by a gravimetric preparation method. The expanded uncertainty in the certified value is equal to $U = ku_c$, where u_c is the combined standard uncertainty derived from the analytical results, the method-to-method variance, the sample homogeneity, the standard solution and dry mass correction, with coverage factor ($k=2$) corresponding to a confidence interval of 95 %.

Metrological Traceability

The certified values were determined with JCSS (Japan Calibration Service System) standard solutions and are traceable to the International System of Units (SI).

Reference Value

The concentration of Co in this CRM is given in the following table as non-certified value. It is expressed in mass fractions after dry mass correction. The expanded uncertainties were determined using a coverage factor of $k = 2$, which is corresponding to an estimated confidence interval of approximately 95 %. The sample pretreatment, dry mass correction, homogeneity assessment, and uncertainty evaluation were carried out in the same way as those for certified values.

Elements	Non-certified 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

Expiration of Certification

The certification of this CRM is valid until March 31, 2030, provided that the material is unopened and stored in accordance with the instructions given in this certificate.

Sample Form

This CRM was prepared from fresh tea leaves, which were powdered by freeze-pulverization after washing and drying. The CRM is in dark brown powder form, which was bottled into an amber glass bottle (ca. 20 g each).

Homogeneity

The homogeneity of this CRM was determined by analyzing 10 bottles hierarchical-randomly selected from the 732 bottles. The trace elements were determined by ICP-MS or ICP-AES after microwave assisted acid digestion. The homogeneity is reflected to the uncertainty of the certified values.

Precautions for Storage

This CRM should be kept in a clean place at room temperature (ca. 25 °C) and shielded from light.

Precautions for Use

- 1) Attention should be paid to avoid contamination when the bottle was 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 0.5 g is recommended for quantification.
- 3) Dry mass correction is required when the CRM is analyzed. The correction factor was obtained by the following procedure.
 - ① Approximately 0.5 g of the CRM is quickly weighted into a weighting glass vessel. The CRM in the vessel is heated at 85 °C for 15-25 hours.
 - ② Weigh the CRM with the vessel after cooling for 30-min in a silica gel desiccator.
 - ③ The difference of the mass before and after drying is assumed as moisture content.

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

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. 7.4 %.

3) Be careful of the following points when the CRM is weighed.

① Do not weigh in a high humidity condition.

② Weighting have to be done as quickly as possible.

③ Do not keep the bottle open.

④ Dry mass correction have to be done parallel to every digestion and analysis.

Precautions for Handling

This CRM is for laboratory use only. This CRM is not edible, and do not eat. Be care 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 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 placed into amber glass bottles (20 g each) by using a split method and was sterilized with γ -ray irradiation (^{60}Co , 20 kGy). The bottles were sealed individually in polypropylene packages and are stored at room temperature.

Reference Value

The concentrations of Ti, V, Cr, Y, and lanthanides were summarized in the follow table as information value. 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 these reference information.

Elements	Reference information, 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

NMLJ Analysts

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

Technical Information

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

Customer registration on the NMIJ WEB site shown below will facilitate notification of above revision. Technical report about this CRM can be also obtained from the contact shown below.

Reproduction of Certificate

In reproducing this certificate, it should be clearly indicated that the document is a copy.

Additional Statement

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

December 22, 2010

Tamotsu Nomakuchi
President

National Institute of Advanced Industrial Science and Technology

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