

National Centre for Compositional Characterisation of Materials (NCCCM),

Bhabha Atomic Research Centre (BARC) Hyderabad, India

Certified Reference Material Major and Minor Constituents in Tea Powder Reference Material Certificate

BARC-D3201

Certified Reference Material (CRM) of tea for major and minor constituents (K, Ca, P, Mg, Mn, Al, Fe, Ba, Zn, Cu, Sr, Pb, As, Cd, and Hg) is intended for use as a calibration standard in evaluating analytical methods and the performance of instruments for the determination of elements. This CRM can also be used for data quality control in the routine analysis of tea powder. One bottle of this CRM contains ~ 20 g of the tea powder in an amber colour glass bottle.

The tea powder material for major and minor constituents has been certified by the consensus of a network of laboratories by means of an inter laboratory comparison exercise (ILCE) as given in table below. The results are referred to the dry material (1g) corrected for moisture at 85°C for 5 h. Analytical techniques used for the determination of major and minor constituents in tea powder by the participant laboratories include inductively coupled plasma atomic emission spectroscopy (ICP-AES), graphite furnace atomic absorption spectroscopy (GFAAS), inductively coupled plasma mass spectroscopy (ICP-MS), atomic fluorescence spectroscopy (AFS), and Hydride Generation Atomic Absorption Spectrophotometry (HGAAS). This tea certified reference material (CRM) was produced in accordance with the ISO 17034: 2016 and ISO/IEC 17025:2017. Assigned property values were established according to ISO Guide 35:2017 guidelines.

Analyte	Certified Values ¹	Expanded uncertainty ² (k =2)	Units
Ca	0.458	0.025	% m/m
K	1.99	0.139	% m/m
Р	0.303	0.031	% m/m
Mg	0.225	0.014	% m/m
Mn	1150	75.0	mg kg ⁻¹
Al	1033	94.0	mg kg ⁻¹
Fe	431	29.5	mg kg ⁻¹
Ba	44.7	2.3	mg kg ⁻¹
Zn	25.5	1.1	mg kg ⁻¹
Cu	19.1	0.79	mg kg ⁻¹
Sr	15.3	1.27	mg kg ⁻¹
Pb	3.45	0.31	mg kg ⁻¹
Cd	1.27	0.11	mg kg ⁻¹
Hg	0.60	0.084	mg kg ⁻¹
As*	1.45	0.36	mg kg ⁻¹

The given uncertainty of the certified value is at a confidence level 95% (Coverage factor k = 2)

¹ ISO 13528 (2015): Statistical methods for use in proficiency testing by inter-laboratory comparison, ²ISO 35: 2017 guidelines, * Indicative value

Origin and preparation of the tea CRM material

Tea granules used for the preparation of candidate tea CRM were procured from local market. A total of 3.6 kg processed material was obtained and packed in 146 glass bottles each containing ~20 g of material.

Homogeneity study

Minimum sample size used for analytical homogeneity is ~500 mg for all the analytes. Bulk homogeneity was established prior to packing the tea powder into 146 bottles. Within and between bottles homogeneity tests for K, Ca, P, Mg, Mn, Al, Fe, Ba, Zn, Cu, Sr, Pb, As, Cd, and Hg were carried out at National Centre for Compositional Characterisation of Materials (BARC-NCCCM), Hyderabad using a validated method based on microwave digestion followed by ICP-AES, GFAAS and AFS measurement. Homogeneity tests were carried out as per ISO 17034:2016 and ISO Guide 35:2017 and the inhomogeneity as evaluated using one way ANOVA is not significant but the same is incorporated into the uncertainty of the certified values.

Instructions for Handling & storage

This CRM should be stored at 4°C temperature. The withdrawal of this material must be carried out in an appropriate environment with clean spatula and remaining material must not be return to the bottle. The bottle should be shaken well before taking the CRM aliquot sample.

Expiration of Certification

Tea powder sample is stable since last twelve months. The material is expected to be stable for next twenty-four months when stored at 4 °C and its further long term stability study would be checked every six months until stock lasts. This validity may be extended as further evidence of stability becomes available. This certification is nullified if the CRM is damaged, contaminated or modified.

Maintenance of Certification

NCCCM-BARC continuously monitors the certified values of all the properties in the CRM over the period of its certification. If any substantive change occurs due to unforeseen reasons that affect the certification before expiration of certification, NCCCM-BARC will notify to the purchaser immediately by notifying in the website: www.cccm.gov.in

Coordination for this CRM production was carried out by Dr. K. Dash, Dr. Lori Rastogi and Shri A. Durga Prasad of NCCCM-BARC. The processing (grinding, sieving and homogenization) of CRM material was carried out at NCCCM-BARC by Sri A. Durga Prasad, Dr. Lori Rastogi and Dr K. Dash. The data analysis and statistical evaluation for certification has been done by Sri A. Durga Prasad and Dr. Lori Rastogi at NCCCM-BARC.

Traceability

The property values assigned to BARC-D3201 certified reference material are the mass fractions of specified major and trace elements, expressed in % m/m and mg kg⁻¹ respectively. These are obtained after carrying out an interlaboratory comparison exercise (ILCE) where 11 laboratories participated. Evidence on metrological traceability to the SI units of reference materials and calibrators used in the characterization process was provided by all participant laboratories.

- 1. National Centre for Compositional Characterisation of Materials, Bhabha Atomic Research Centre, ECILpost Hyderabad - 500 062
- 2. Analytical Chemistry Division Bhabha Atomic Research Centre Trombay, Mumbai 400 085
- 3. Centre for Materials for Electronics Technology (C MET) IDA Phase III, HCL (Post), Hyderabad 500051
- 4. Bureau Veritas (India) Pvt. Ltd. F-2, Phase III, Thiru Vi Ka Industrial Estate Chennai 600032
- 5. Analytical Chemistry Group Defence Metallurgical Research Laboratory Kanchanbagh, Hyderabad-500058
- 6. Export Inspection Agency Ministry of commerce and Industry, Kolkata 700107
- 7. Export Inspection agency Kochi Laboratory Ministry of Commerce and Industry, Kochi 682036
- 8. FirstSource Laboratory Solutions LLP, IDA, Nacharam, Hyderabad, 500 076
- 9. Indian Institute of Food Processing Technology Ministry of Food Processing Industries (MoFPI), Thanjvur 613005
- 10. National Commodities Management services Limited, IDA –Uppal, Hyderabad 500 039
- 11. SGS India Private Limited, Ambattur Industrial Estate, Chennai 600058

Analytical Method

After shaking the bottle well about 500 mg sample aliquot is recommended to be taken for acid digestion. The recommended sample processing is microwave assisted acid digestion method by conc. nitric acid (2 mL, HNO₃), hydrogen peroxide (3 mL, 30 %, v/v) and 5ml DI water (the microwave digestion step includes a hold time of 30 min at 220 - 230°C). Quantification was carried out using external calibration. Analysis of samples should be done after suitable dilutions. The moisture content should be determined by drying a separate portion of sample at 85°C for 5 hr.

Legal disclaimer

The certified values of major and trace elements given in this certificate are the best estimates of true values within the stated uncertainties and based on the techniques described in this certificate. The certifying organizations, i.e. BARC-NCCCM have taken into account appropriate international guidelines for the preparation and certification of material, however, they assume no liability with respect to, or for damages resulting from, the use of any information, material, apparatus, method or process disclosed in this certificate or any warranties with respect to the material (Pl. see the material safety data sheet) safety and the data contained in this reference sheet and shall not be liable for any damage that may result from the use of such material/ data.

Validity: 30th April, 2026

Signature: Name: Dr. Awadhesh Kumar Affiliation: Associate Director Chemistry Group Bhabha Atomic Research Centre, Mumbai Website: <u>www.cccm.gov.in</u> Contact: 022-25595330