



BARC-National Centre for Compositional  
Characterization of Materials (NCCCM),  
Hyderabad-500062



CSIR-National Physical Laboratory (NPL), New Delhi-110012

## भारतीय निर्देशक द्रव्य Indian Reference Material

**BND 4101.01**

Trace Elements in High Purity Quartz

### *Certificate of Analysis*

This Bharatiya Nirdeshak Dravya (BND) i.e. Certified Reference Material (CRM) of trace elements Aluminium, Iron, Calcium, Sodium, Potassium, Magnesium, and Titanium (Al, Fe, Ca, Na, K, Mg, and Ti) in high purity quartz powder is intended for use as a calibration standard in evaluating analytical methods, instruments performance for the determination of elements. This BND or CRM can also be used for data quality control (DQC) in the routine analysis of quartz. One bottle of BND 4101.01 contains about 25g of the powdered quartz material packaged in a HDPE sealed bottle.

The quartz powder material for its trace element impurities has been certified by NCCCM-BARC and CSIR-NPL by means of an inter-laboratory comparison (ILC) exercise as given in table below. Analytical techniques used for trace elements measurements in quartz by the participant's lab are ICP-OES (Ca, Fe, Mg, Ti, K, Na), FAAS (Al, K, Fe, Mg, Na) and TXRF (Ca). The assigned property values of trace elements were established according to ISO Guide-35: 2006 guidelines.

Sr. No.	Element/ Analyte	Mass fraction <sup>1</sup> (mg kg <sup>-1</sup> )	Expanded uncertainty <sup>2</sup> (mg kg <sup>-1</sup> )
1	Al	1181	96
2	Ca	58	8
3	Fe	81	9
4	K	558	35
5	Mg	20	3
6	Na	249	16
7	Ti	17	3

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

<sup>1</sup>ISO 35: 2006 guidelines

<sup>2</sup>JCGM100:2008 Evaluation of measurement data – Guide to the expression of uncertainty in measurement.

### **ILC Participating laboratories:**

Analytical Chemistry Division, Bhabha Atomic Research Centre (BARC), Trombay, Mumbai  
Analytical Control laboratory, BARC, Mysore-571130  
Centre for Materials for Electronics Technology (C-MET), Cherlapally, Hyderabad-500051  
CSIR-National Physical Laboratory (NPL), Dr. K.S. Krishnan Marg, New Delhi-110012  
Control Laboratory, Nuclear Fuel Complex (NFC), Hyderabad-500062  
National Centre for Compositional Characterization of Materials (NCCCM, BARC),  
Hyderabad-500062

### **Origin and preparation of the material**

A sample of ~10 kg of quartz was collected from quartz mines in the state of Tamil Nadu. The material was milled to a powder in a grinder and then sieved through a 150 micron sieve. The sieved material with a particle size less than 150  $\mu\text{m}$  was further homogenized. Homogeneity was achieved by mixing the material in a polypropylene rotating homogenizer for 24 hours. 25g of aliquot were packed into 350 pre-cleaned high density polyethylene (HDPE) bottles.

### **Homogeneity and Stability study**

Sample processing and homogeneity tests were carried out at National Centre for Compositional Characterization of Materials (NCCCM) Hyderabad, a unit of BARC. Minimum sample size used for analytical homogeneity is ~200 mg for all the analytes. Homogeneity within and between bottles were carried out as per ISO Guide 34 & 35 before despatching the samples to participating laboratories for trace elements ILC/ certification measurement comparison. Quartz powder sample is stable since last two years as seen in earlier testing scheme of a PT. The material is expected to be stable for many years at room temperature and its further long term stability study would continue until stock lasts.

### **Validity and Instructions for Handling and storage**

User should take the precaution to prevent contamination of the material during use or storage. This certificate is valid till 30 April 2018 provided it is used with recommended handling and stored. NCCCM will periodically check for its stability and inform the customer if required. This validity may be extended as further evidence of stability becomes available. The material is quartz-150 mesh (silicon dioxide) powder containing about 25g of the material in HDPE bottle. The BND bottle may be stored at room temperature in safe custody and clean environment. This certification is nullified if the BND is damaged, contaminated or modified.

### **Maintenance of Certification**

BARC-NCCCM continuously monitors the certified value of trace elements in the BND over the period of its certification. If any substantive change occurs due to unforeseen reasons that affect the certification before expiration of certification, BARC-NCCCM will notify to the purchaser immediately.

Coordination for this BND preparation and certification was done by Dr. Sunil Jai Kumar (BARC-NCCCM) under guidance of Dr B.N. Jagatap, Director Chemistry Group and Prabhat K. Gupta (CSIR-NPL). CRM was processed at BARC-NCCCM by Dr Kulamani Dash. The data analysis and statistical evaluation for certification has been done by Dr. Daya Soni (CSIR-NPL) and Dr K Chandrasekaran (BARC-NCCCM). Other contributors are Dr. Shankar G. Aggarwal (CSIR-NPL) and Shri S.Thangvel (BARC-NCCCM).

## Traceability

The quantity values assigned to the BND 4101.01 certified reference material are the mass fractions of specified trace elements, expressed in the derived SI unit  $\text{mg kg}^{-1}$ . Evidence on metrological traceability to the SI units of reference materials and calibrators used in the characterization process was provided by all participant laboratories.

## Analytical Method

Quartz powder sample is dried @  $\sim 110^\circ\text{C}$  for two hours (negligible moisture content,  $\sim < 0.1\%$ ) and about 200 to 300 mg sample aliquot is recommended to be taken for acid digestion. The participating labs were free to choose any suitable method. The recommended sample processing acid digestion method was by hydrofluoric acid (HF) and conc. nitric acid ( $\text{HNO}_3$ ) and thereafter, the silicon matrix was evaporated as  $\text{SiF}_4$  and trace analyte residues were taken in 5% (v/v) nitric acid prior to quantification using external calibration. Results were reported at dry mass basis.

## Legal disclaimer

The certified values of 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 and CSIR-NPL 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.

*Sunil Jai Kumar*  
Signature: 01/02/2016  
Name: Dr Sunil Jai Kumar  
Affiliation: Head, (BARC-NCCCM)  
Website: [www.cccm.gov.in](http://www.cccm.gov.in)  
Contact: 040-27121365

*Prabhat K. Gupta*  
Signature: 01/02/2016  
Name: Prabhat K. Gupta  
Affiliation: Chief Scientist, CSIR-NPL  
Website: [www.nplindia.org](http://www.nplindia.org)  
Contact: 011-45608441, 85608373

*Dr B N Jagatap*  
Signature: 01/02/2016  
Name: Dr B N Jagatap  
Affiliation: Director, Chemistry Group (BARC-NCCCM)  
Website: [www.cccm.gov.in](http://www.cccm.gov.in)  
Contact: 022-25595056

**Material Safety Data Sheet (MSDS) for Quartz Powder**

**HAZARDS IDENTIFICATION**

**NFPA Ratings (Scale 0–4):** Health = 1 Fire = 0 Reactivity = 0

**Major Health Hazards:** Cancer (in humans) (a)

**Physical Hazards:** There are no known physical hazards associated with this material.

**Potential Health Effects (Acute and Chronic):**

**Inhalation:** Acute: mechanical irritation; chronic: same as acute and chest pain, weight loss, difficulty breathing, digestive disorders, bluish skin colour, lung damage, cancer, and death.

**Skin Contact:** Mechanical irritation.

**Eye Contact:** Mechanical Irritation and possible eye damage.

**Ingestion:** No information available on severe effects.

**Listed as a Carcinogen/Potential Carcinogen: Yes**

In the National Toxicology Program (NTP) Report on Carcinogens X(a)

In the International Agency for Research on Cancer (IARC) Monographs X(b)

By the Occupational Safety and Health Administration (OSHA) X

(a) NTP lists silica, crystalline, as a known carcinogen.

(b) IARC lists silica (quartz) as a Group 1 (carcinogenic to humans).



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Indian Reference Material**

ISO guide 34  
accredited  
Certificate no:  
RC-1007

**BND 4101.01**

**Trace Elements in High Purity Quartz**

Date: 30 April 2018

Addendum

Extension of Validity: Based on the stability tests carried out by National Centre for Compositional Characterisation of Materials, the validity of this certificate has been extended to 30<sup>th</sup> April 2023.

*Sunil jai Kumar*

Authorised Signatory

Dr. Sunil Jai Kumar

Head, NCCCM, ECIL-post

Hyderabad – 500 062

India