



**National Centre for Compositional Characterisation
of Materials (NCCCM),
Bhabha Atomic Research Centre, Hyderabad, India
and
Atomic Minerals Directorate for Exploration and
Research (AMDER), Hyderabad, India**

**Certified Reference Material (CRM)
Major and Minor Constituents in Dolomite
Certificate of Analysis**

CRM BARC B1101

सीआरएम बीएआरसी बी ११०१

Certified Reference Material (CRM) of dolomite for major and minor constituents (Al_2O_3 , BaO , CaO , Fe_2O_3 , MgO and SrO) 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 (DQC) in the routine analysis of dolomite. One bottle of BARC B1101 contains 50g of the powdered dolomite material packaged in a sealed HDPE bottle.

The dolomite powder material has been certified for major and minor constituents by NCCCM-BARC and AMDER by means of an inter laboratory comparison exercise (ILCE) as given in table below. The results are certified on the material dried at 105°C . Analytical technique used for the determination of major and minor constituents by the participant laboratories is ICP-OES. The certified reference material of dolomite was prepared in accordance with the ISO Guide 17034:2016 and ISO IEC 17025:2017. The assigned property values were established according to ISO Guide 35:2017/ ISO 13528:2015 guidelines.

Constituent	Unit	Content ^{1,2}	Expanded uncertainty ^{1,2}
Al_2O_3	(wt.%)	1.3	0.1
BaO	(mg/kg)	1121	82
CaO	(wt.%)	26.9	1.0
Fe_2O_3	(wt.%)	1.1	0.1
MgO	(wt.%)	18.7	0.4
SrO	(mg/kg)	126	9

The given uncertainty of the certified value is at a confidence level 95% (coverage factor $k=2$)
¹ISO 35: 2017 guidelines, ² ISO 13528 (2015): Statistical methods for use in proficiency testing by inter-laboratory comparison.

Preparation of CRM of Dolomite

a) Origin and preparation of the material

Approximately ~10kg of dolomite was collected from the uranium mines at Tummalapalle, YSR District, Andhra Pradesh. The crude material was crushed, milled and sieved to a powder of around 10 micron particle size. The sieved material was further homogenized through a mechanical homogenizer. The above processes were carried out at Atomic Minerals Directorate of Exploration and Research (AMDER), Hyderabad. Fifty (50) g portion each of the certified material was packed into 150 Nos. of pre-cleaned high density polyethylene (HDPE) bottles.

b) Homogeneity of dolomite powder and stability study

Sample preparation for analysis and homogeneity tests were carried out at National Centre for Compositional Characterisation of Materials (NCCCM) Hyderabad, a unit of BARC. Minimum sample size used for analytical homogeneity is ~250mg for all the analytes. Bulk homogeneity was established prior to packing the dolomite powder into bottles. Homogeneity with respect to between bottles was ensured and the minimum sample needed to maintain analytical homogeneity was established. These exercises were carried out as per ISO Guide 17034:2016 and ISO Guide 35:2017 before despatching the samples to the participating laboratories for ILCE for six properties (Al_2O_3 , BaO, CaO, Fe_2O_3 , MgO and SrO). Due to its geological origin dolomite is expected to be stable at ambient temperature for 5 years from the date of its release and studies on its further long term stability would continue until stock lasts.

c) Validity and Instructions for Handling and storage

User should take precautions to prevent contamination of the material during use or storage. The material, dolomite powder (50g, ~ 10micron) is supplied in HDPE bottle. The CRM bottle may be stored at ambient temperature in safe custody and clean environment. This certificate is valid for 5 years from the date of release provided it is handled and stored as mentioned above. NCCCM will periodically check for its stability and inform the customer if required. This validity may be extended as evidence of further stability becomes available. This certification is nullified if the CRM is damaged, contaminated or modified.

d) Maintenance of Certification

NCCCM-BARC continuously monitors the certified value 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.

Coordination for this CRM preparation and certification was done by Dr. Sanjiv Kumar, Head, NCCCM-BARC under the guidance of Dr. A. K. Tyagi, Director, Chemistry Group, BARC and

Dr. D. K. Sinha, Director, AMDER. The CRM preparation was initiated by Dr. N. Satyanarayana and was processed at NCCCM-BARC by Dr. A. C. Sahayam and Shri G. Venkateswarlu. Analytical aspects and sample pre-treatment was coordinated and guided by Dr. A. A. Patwardhan, Head, Chemistry Group, AMDER. The data analysis and statistical evaluation for certification has been done by Dr. K. Chandrasekaran (NCCCM-BARC).

e) Traceability

The property values assigned to BARC B-1101 certified reference material are the mass fractions of specified major (wt.%) and trace elements, expressed in the derived SI unit mg/kg. Evidence on metrological traceability to the SI units of reference materials and calibrators used in the characterization process was provided by all participant laboratories.

f) Analytical Method

Dolomite powder is dried at $\sim 105^{\circ}\text{C}$ for two hours (negligible moisture content, $< 0.01\%$) and about 0.250 to 0.500g sample portion is recommended to be taken for the analysis. The participating laboratories were free to choose any suitable method. The recommended sample processing method is as described in ASTM C1301-95:2014. The sample (0.250g) is fused with 1.5g lithium metaborate and the clear melt is dissolved in HNO_3 . The sample solution is analysed by ICP-OES after suitable dilutions. Wavelengths of measurements against each property are: Al: 396.152nm, Ba: 455.403nm, Ca: 317.933nm, Mg: 279.079nm, Fe: 259.940nm and Sr: 407.771nm. Quantifications are carried out using external calibration. Results are reported on dry mass basis.

Participating laboratories in the Inter Laboratory Comparison Exercise (ILCE):

Analytical Chemistry Division (Chemical Method Section), Bhabha Atomic Research Centre (BARC), Trombay, Mumbai

Analytical Chemistry Division (Analytical Spectroscopy Section), Bhabha Atomic Research Centre (BARC), Trombay, Mumbai

Analytical Control Laboratories, BARC, Mysore

Centre for Materials for Electronics Technology (C-MET), Cherlapally, Hyderabad

Chemistry Lab, Atomic Minerals Directorate for Exploration and Research, Bengaluru

Chemistry Lab, Atomic Minerals Directorate for Exploration and Research, Hyderabad

Chemistry Lab, Atomic Minerals Directorate for Exploration and Research, Jaipur

Chemistry Lab, Atomic Minerals Directorate for Exploration and Research, Nagpur

Chemistry Lab, Atomic Minerals Directorate for Exploration and Research, New Delhi

Chemistry Lab, Atomic Minerals Directorate for Exploration and Research, Shillong

Control Laboratory, Nuclear Fuel Complex (NFC), Hyderabad

Fuel Chemistry Division, Bhabha Atomic Research Centre (BARC), Trombay, Mumbai

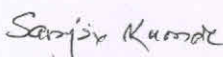
Mineral Processing Division, Bhabha Atomic Research Centre (BARC), Hyderabad

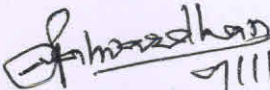
National Centre for Compositional Characterisation of Materials (NCCCM, BARC), Hyderabad


SGS India Private Limited, Chennai


Legal disclaimer

The certified values of major and trace constituents 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 organisations, i.e. NCCCM-BARC and AMDER 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.

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Affiliation: Director, AMDER
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Material Safety Data Sheet (MSDS) for Dolomite Powder

HAZARDS IDENTIFICATION

HMIS Ratings (Scale 0–4): Health = 1, Fire = 0, Reactivity = 0

Hazard scale: 0 = Minimal, 1 = Slight, 2 = Moderate, 3 = Serious, 4 = Severe

Major Health Hazards: Cancer (in humans)

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

Potential Health Effects (Acute and Chronic):

Inhalation: Long term exposure causes pulmonary fibrosis.

Skin Contact: Skin irritation.

Eye Contact: Irritation to eye tissues.

Ingestion: Temporary irritation to throat, stomach and gastrointestinal tract.

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

Dolomite contains crystalline silica in the form of quartz,

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

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

Bottle cover

The bottle cover will bear the following label as agreed upon by the collaborating laboratories, NCCCM (BARC) and AMDER.



Where;

BARC is organisation name

B is inorganic material

1 is minerals and ores

1 is CRM serial number

01 is batch number of particular CRM

Intended and instructions for use

This Certified Reference Material (CRM) is intended for use in evaluating analytical methods, instruments performance for the determination of major and trace elements (CaO, MgO, Al₂O₃, Fe₂O₃, BaO and SrO). This CRM can be used for data quality control (DQC) in the routine analysis of dolomite. Sample processing and homogeneity tests were carried out at National Centre for Compositional Characterization of Materials (NCCCM) Hyderabad, a unit of BARC. This certificate is valid till 11/2026 (5 yrs). This validity may be extended as further evidence of stability becomes available. Minimum sample size for analytical homogeneity is 250 mg for all analytes.