



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



**प्रमाणित निर्देशक द्रव्य
Certified Reference Material
Major and Rare Earth Elements (REEs) in Ferrocarbonatite**

**BARC-B1401
Reference Material Certificate**

Certified Reference Material (CRM) of ferrocarbonatite for major (Al, Ca, Fe, Mg, Mn and P) and rare earth elements (Ce, Dy, Er, Eu, Gd, La, Nd, Pr, Sc, Sm, Tb, Y and Yb) is intended for use as a calibration standard in evaluating analytical methods, the performance of instruments for the determination of elements and for data quality control (DQC) in the routine analysis of ferrocarbonatite. This CRM can also be used by industries which are involved in extraction of rare earth elements from ferrocarbonatite. One unit of BARC-B1401 contains ~100 g of the powdered ferrocarbonatite material packed in a glass bottle. The ferrocarbonatite powder material has been certified for major elements and REEs by NCCCM-BARC and AMDER by means of an interlaboratory comparison exercise (ILCE). The certified values along with expanded uncertainties are given in the table. Analytical techniques used for the determination of major and rare earth elements by the participant laboratories are ICP-OES and ICP-MS. This ferrocarbonatite CRM was produced in accordance with the ISO 17025:2017, ISO 17034:2016 and ISO Guide 35:2017.

Certified mass fraction values for BARC-B1401

S.No.	Analyte	Unit	Mass fraction ¹	± Expanded uncertainty ² (k = 2)
1	Al	% m/m	0.64	0.03
2	Ca	% m/m	15.3	0.4
3	Fe	% m/m	7.5	0.4
4	Mg	% m/m	7.1	0.2
5	Mn	% m/m	1.66	0.08
6	P	% m/m	0.58	0.02
7	Ce	mg/kg	6083	255
8	Dy	mg/kg	41.8	3.3
9	Er	mg/kg	16.4	1.4
10	Eu	mg/kg	35.2	1.1
11	Gd	mg/kg	92	3
12	La	mg/kg	4260	168
13	Nd	mg/kg	1422	42
14	Pr	mg/kg	502	15
15	Sc	mg/kg	18.2	1.3
16	Sm	mg/kg	173	12
17	Tb	mg/kg	12.4	2.2
18	Y	mg/kg	175	7
19	Yb	mg/kg	10.6	1.0

¹ISO 13528:2022 (E): Statistical methods for use in proficiency testing by interlaboratory comparison,

²ISO Guide 35:2017

Indicative mass fraction values for BARC-B1401

S.No.	Analyte	Unit	Mass fraction ¹
1	Ho	mg/kg	8.0
2	Lu	mg/kg	1.2
3	Tm	mg/kg	2.5

¹ISO 13528:2022 (E): Statistical methods for use in proficiency testing by interlaboratory comparison

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

Chemistry Laboratory, Atomic Mineral Directorate for Exploration and Research, Hyderabad
Chemistry Laboratory, Atomic Mineral Directorate for Exploration and Research, Jaipur
Chemistry Laboratory, Atomic Mineral Directorate for Exploration and Research, Nagpur
Chemistry Laboratory, Atomic Mineral Directorate for Exploration and Research, Bengaluru
Chemistry Laboratory, Atomic Mineral Directorate for Exploration and Research, New Delhi
Chemistry Laboratory, Atomic Mineral Directorate for Exploration and Research, Jamshedpur
Chemistry Laboratory, Atomic Mineral Directorate for Exploration and Research, Shillong
NALCO Research and Technology Centre (NRTC-NALCO), Bhubaneswar
Control Laboratory, Nuclear Fuel Complex (NFC), Hyderabad
Mineral Processing Division, Bhabha Atomic Research Centre (BARC), Hyderabad
National Centre for Compositional Characterisation of Materials (NCCCM-BARC), Hyderabad

Origin and preparation of the material

Approximately 20 kg of candidate ferrocarnatite CRM was collected from Amba Dongar, Gujarat by AMDER and was crushed, milled to powder form at their Hyderabad facility. It was dried and sieved to a powder of around $\leq 180 \mu\text{m}$ particle size at NCCCM-BARC, Hyderabad. The sieved material was homogenized using a mechanical homogenizer. This was packed into 175 pre-cleaned glass bottles each containing ~100 g of material.

Homogeneity assessment and ILCE

Homogeneity assessment of candidate ferrocarnatite CRM at different stages were carried out at NCCCM-BARC, Hyderabad. Minimum sample size used for analytical homogeneity is ~200 mg for all the analytes. Bulk homogeneity was established prior to packing the candidate ferrocarnatite CRM into bottles. After establishing bulk homogeneity of the material, assessment of within and between bottle homogeneity was also carried out. The homogeneity with respect to all property values were established using one way ANOVA as per ISO 17034:2016 and ISO Guide 35:2017. The uncertainty due to inhomogeneity was incorporated for calculating combined uncertainty of the certified values. After establishing within and between unit homogeneity, interlaboratory comparison exercise (ILCE) was conducted for the certification of nineteen property values which included major elements (Al, Ca, Fe, Mg, Mn and P) and REEs (Ce, Dy, Er, Eu, Gd, La, Nd, Pr, Sc, Sm, Tb, Y and Yb).

Instructions for use, handling & storage

A minimum of ~200 mg sample should be used for the analysis of major elements (Al, Ca, Fe, Mg, Mn and P) and REEs (Ce, Dy, Er, Eu, Gd, La, Nd, Pr, Sc, Sm, Tb, Y and Yb) in this CRM. The CRM bottle should be stored at room temperature. The withdrawal of this material from the bottle must be carried out in an appropriate environment with clean spatula and remaining material must not be returned to the bottle. Ferrocarnatite CRM (BARC-B1401) is supplied in glass bottle containing ~100 g material.

Expiration of certification

The certificate of this CRM is valid for 10 years from the date of release, within the uncertainty specified, provided the CRM is handled and stored in accordance with the instructions given in this certificate. NCCCM-BARC will periodically check (every 2 years) for the stability and inform the customer if required. 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 till the stock lasts. If any substantive change occurs due to unforeseen reasons that affect the certification before expiration of certificate, NCCCM-BARC will notify to the purchasers through the website: <https://cccm.gov.in>

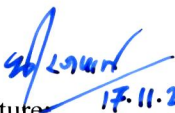
Coordination for this CRM preparation and certification was done by Dr. K. Dash, Head, NCCCM-BARC and Dr. S. Durani, Head, Chemistry Group, AMDER, Hyderabad under the guidance of Dr. A. C. Bhasikuttan, Associate Director, Chemistry Group, BARC, Mumbai and Shri Dheeraj Pande, Director, AMDER, Hyderabad. The CRM preparation was carried out by Shri S. Thangavel, Shri G. Venkateswarlu, Ms. Lisa Nayak, Dr. K. Dash from NCCCM-BARC and Dr. Beena Sunil Kumar, Dr. Johnson George, Dr. S. Durani from AMDER, Hyderabad. The data analysis and statistical evaluation for certification has been done by Shri A. Durga Prasad and Ms. Lisa Nayak from NCCCM-BARC.

Traceability

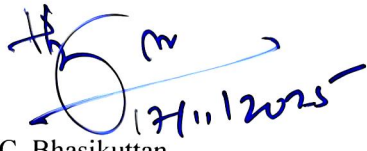
The property values, assigned to BARC-B1401 CRM, are expressed in % mass fraction for major elements and in mg/kg for REEs. These values were assigned after carrying out an interlaboratory comparison exercise (ILCE) where 11 laboratories participated. Evidence on metrological traceability to the SI units of reference materials and calibrators used in the characterization process was provided by all participant laboratories.

Legal disclaimer

The certified values of major and rare earth 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.*, 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.

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