



National Centre for Compositional Characterization of  
Materials (NCCCM), Bhabha Atomic Research Centre  
(BARC) Hyderabad, India  
National Aluminium Company Limited (NALCO)  
Bhubaneswar, India



### प्रमाणित निर्देशक द्रव्य

#### Certified Reference Material

#### Major and Minor Constituents in Bauxite

#### Reference Material Certificate

### BARC B1201

Certified Reference Material (CRM) of bauxite for major and minor constituents ( $\text{Al}_2\text{O}_3$ ,  $\text{Fe}_2\text{O}_3$ ,  $\text{SiO}_2$ ,  $\text{TiO}_2$ ,  $\text{Cr}_2\text{O}_3$ ,  $\text{MgO}$ ,  $\text{MnO}$ ,  $\text{V}_2\text{O}_5$  and LOI) is intended for use as a calibration standard in evaluating analytical methods and the performance of instruments for the determination of analytes. This CRM can also be used for data quality control (DQC) in the routine analysis of bauxite. One bottle of this CRM contains 90g of the powdered bauxite material in a polypropylene bottle.

The bauxite 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. Analytical techniques used for the determination of major and minor constituents in bauxite by the participant laboratories include inductively coupled plasma atomic emission spectroscopy (ICP-AES), Titrimetry, XRF, Gravimetry and Thermogravimetry analysis (TGA). This bauxite certified reference material (CRM) was produced in accordance with the ISO GUIDE 34: 2016 and ISO IEC 17025:2005. Assigned property values were established according to ISO Guide 35:2017 guidelines. The results are referred to the dry material (1g) corrected for moisture at 105°C for 2 h.

Analyte	Certified Values <sup>1</sup> Mass Fraction (% m/m)	Expanded uncertainty <sup>2</sup> (% m/m)
$\text{Al}_2\text{O}_3$	41.3	0.5
$\text{Fe}_2\text{O}_3$	29.3	0.5
$\text{SiO}_2$	4.12	0.23
$\text{TiO}_2$	2.33	0.05
$\text{Cr}_2\text{O}_3$	0.044	0.003
$\text{MnO}$	0.057	0.005
$\text{MgO}$	0.027	0.003
$\text{V}_2\text{O}_5$	0.070	0.003
LOI @1000°C	22.3	0.2

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

<sup>1</sup> ISO 13528 (2015): Statistical methods for use in proficiency testing by inter-laboratory comparison, <sup>2</sup>ISO 35: 2017 guidelines,

#### Additional information

The following four property values (mean and standard deviation) are indicative derived from the homogeneity test of the material from the ten bottles.

Property values	Unit	Mass fraction $\pm$ SD	Analytical method
Ga <sub>2</sub> O <sub>3</sub>	% m/m	0.0094 $\pm$ 0.0005	ICP-MS
ZnO	% m/m	0.008 $\pm$ 0.001	ICP-MS
CaO	% m/m	0.009 $\pm$ 0.001	ICP-AES
Extractable Alumina <sup>1</sup>	% m/m	34.2 $\pm$ 0.4	Titrimetry

<sup>1</sup> amount of alumina that is digested in a caustic solution (alumina to caustic ratio ~0.9) @ 145°C following conditions of Bayer Process.

### Origin and preparation of the CRM bauxite material

The original bauxite material was mined from the NALCO mines at Damanjodi, Odisha, India by the R&D department of Mines & Refinery. The material was dried at 105°C for 48 hours. The dried material is ground by cross beater mill (SK300) and sieved through 106 $\mu$  sieve. A total of 14.4 kg processed material (<106 $\mu$ ) was obtained and packed in 156 polypropylene bottles each containing 90g.

### Homogeneity study

Minimum sample size used for analytical homogeneity is ~200 mg for all the analytes. Bulk homogeneity was established prior to packing the bauxite powder into 156 bottles. Within and between bottles homogeneity tests for Al<sub>2</sub>O<sub>3</sub>, Fe<sub>2</sub>O<sub>3</sub>, SiO<sub>2</sub>, TiO<sub>2</sub>, Cr<sub>2</sub>O<sub>3</sub>, MgO, MnO, V<sub>2</sub>O<sub>5</sub> were carried out at National Centre for Compositional Characterization of Materials (BARC-NCCCM), Hyderabad using a validated method based on microwave digestion followed by ICP-AES measurement. The homogeneity tests for loss on ignition (LOI) were carried at the R&D Department, Mines & Refinery complex of NALCO at Damanjodi, Koraput, Odisha. Homogeneity tests were carried out as per ISO Guide 34:2016 and ISO Guide 35:2017 (en) and the inhomogeneity as evaluated using ANOVA is not significant but the same is incorporated into the uncertainty of the certified values.

### Instructions for use, handling & storage

A minimum of 0.2g sample should be used for analysis. A separate sample aliquot (1g) must be taken for moisture correction at 105  $\pm$  2 °C for 2 hours. This CRM should be stored at room temperature in clean space to prevent contamination of the material. 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 five times before taking the CRM aliquot sample.

### Expiration of Certification

Due to its geological origin bauxite is expected to be stable for 10 years from the date of its release February, 2022 and its further long term stability study would continue at regular intervals (2 years) until stock lasts. This certificate is valid till 30<sup>th</sup> January 2031, provided it is handled and stored in accordance with the recommended protocols. 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. 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](http://www.cccm.gov.in)

Coordination for this CRM production was carried out by Dr. K. Dash (NCCCM-BARC), Mrs V. Krishnakumari and Sri Vinod Kumar Verma of the R&D Department, Mines & Refinery Complex of NALCO. The processing (grinding, sieving and homogenization) of CRM material was carried out at NCCCM-BARC by Sri A. Durgaprasad, Dr. Lori Rastogi and Dr K. Dash. The data analysis and statistical evaluation for certification has been done by Sri A. Durgaprasad, Dr. Lori Rastogi and Sri S. Thangavel at NCCCM-BARC.

### Traceability

The property values assigned to BARC-B1201 certified reference material are the mass fractions of specified major and trace analytes, expressed in % m/m and these are obtained after carrying out an interlaboratory comparison exercise (ILCE) where 17 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.

#### **ILCE Participating laboratories**

Analytical chemistry Division, Bhabha Atomic Research Centre, Trombay, Mumbai  
Chemistry Lab, Atomic Minerals Directorate for Exploration and Research, DAE, Bengaluru.  
Chemistry Lab, Atomic Minerals Directorate for Exploration and Research, DAE, Hyderabad.  
Chemistry Lab, Atomic Minerals Directorate for Exploration and Research, DAE, Jaipur.  
Chemistry Lab, Atomic Minerals Directorate for Exploration and Research, DAE, Nagpur.  
Chemistry Lab, Atomic Minerals Directorate for Exploration and Research, DAE, New Delhi.  
Chemistry Lab, Atomic Minerals Directorate for Exploration and Research, DAE, Jamshedpur.  
Control Laboratory, Nuclear Fuel Complex (NFC), DAE, Hyderabad-500062.  
CSIR-Institute of Minerals & Materials Technology, IMMT Campus, Bhubaneswar, Odisha-751013.  
Jawaharlal Neheru Aluminium research and Design Centre, Amaravati Road, Wadi, Nagpur-440023.  
Lucid Laboratories, Balanagar, Hyderabad, India-500037.  
NALCO Research & Technology Centre, Bhubaneswar, Odisha, India-751003.  
NALCO R&D Department, Mines & Refinery Complex, Damanjodi, Koraput, Odisha, India-763008.  
National Centre for Compositional Characterization of Materials (BARC-NCCCM), DAE, Hyderabad-500062.  
SGS India Private Limited, Ambattur Industrial Estate, Chennai, India-600058.  
SGS India Private Limited, Mancheswar Industrial Estate, Bhubaneswar, Odisha, India-751010.  
Utkal Alumina International, Laboratory, Rayagada, Odisha 765001.

#### **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 and NALCO 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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