



**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

Reference Material Certificate for Trace Impurities in Alumina

BARC- B1301

Certified Reference Material (CRM) of alumina for trace impurities (Na_2O , CaO , TiO_2 , V_2O_5 , Fe_2O_3 and Ga_2O_3) 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) material in the routine analysis of alumina. One bottle of this CRM contains ~ 85 g of the alumina powder material in a polypropylene bottle.

Alumina powder has been certified for trace impurities 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 trace impurities in alumina by the participant laboratories include inductively coupled plasma atomic emission spectrometry (ICP-AES), inductively coupled plasma mass spectrometry (ICP-MS) and X-ray fluorescence (XRF). This alumina certified reference material (CRM) was produced in accordance with the ISO 17025:2017, ISO 17034:2016 and ISO Guide 35: 2017. The results are referred to the dry material (1g) corrected for moisture at 105°C for 2 h.

Analyte	Certified Values ¹ Mass Fraction (% m/m)	Expanded uncertainty ² (% m/m)
Na_2O	0.271	0.012
CaO	0.031	0.005
TiO_2	0.0047	0.0005
V_2O_5	0.0014	0.0001
Fe_2O_3	0.0099	0.0008
Ga_2O_3	0.0057	0.0006

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

¹ISO 13528 (2015): Statistical methods for use in proficiency testing by inter-laboratory comparison

²ISO Guide 35: 2017

Additional information

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

Property values	Unit	Mass fraction $\pm 2.SD$	Analytical method
MgO	% m/m	0.0011 \pm 0.0003	ICP-AES
MnO	% m/m	0.0003 \pm 0.0001	ICP-MS
ZnO	% m/m	0.0008 \pm 0.0003	ICP-MS
Loss On Ignition (300 - 1000°C)	% m/m	1.06 \pm 0.04	Thermogravimetry
Surface area	m ² /gm	71.7 \pm 1.6	BET

Origin and preparation of the CRM alumina material

The original alumina material (~ 20 kg) was obtained from the alumina refinery of NALCO at Damanjodi, Odisha, India. The obtained material was sieved through 106 μ m sieve. A total of 13.2 kg processed material (<106 μ m) was obtained and packed in 154 polypropylene bottles each containing 85 g of alumina.

Homogeneity study

Minimum sample size used for analytical homogeneity is ~100 mg for all the analytes. Within and between bottles homogeneity tests for Na₂O, MgO, CaO, TiO₂, V₂O₅, MnO, Fe₂O₃, ZnO and Ga₂O₃ were carried out at National Centre for Compositional Characterization of Materials (NCCCM-BARC), Hyderabad and at NALCO Research and Technology Centre – Bhubaneswar using a validated method based on microwave digestion followed by ICP-AES and ICP-MS measurements. The homogeneity tests for loss on ignition (LOI) and surface area were carried out using thermogravimetry and Brunauer-Emmett-Teller method (BET) analysis respectively at NRTC Bhubaneswar NALCO. Homogeneity tests were carried out as per ISO 17034:2016 and ISO Guide 35:2017 (en) and the inhomogeneity as evaluated using one way ANOVA is not significant but the same is incorporated into the uncertainty of the certified values. Dr. Lori Rastogi, Shri Ankam Durga Prasad, Shri A. Khuntia, Shri L. Naresh and Shri S. Thangavel carried out the homogeneity test.

Instructions for use, handling & storage

A minimum of 0.1 g sample should be used for the 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 sample aliquot.

Expiration of Certification

Alumina, being a highly refractory in nature, is expected to be stable for 20 years from the date of its release August, 2024. Further, its long-term stability study would continue at regular intervals (2 years) until stock lasts. This certificate is valid till 30th August 2044, 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 certificate, 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 (NCCCM-BARC) and Dr. S. P. Mahopatra of NRTC-NALCO. The processing (sieving and homogenization) of CRM material was carried out at NCCCM-BARC by Sri A. Durga Prasad and Dr. Lori Rastogi. 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-B1301 certified reference material are the mass fractions of specified trace impurities, expressed in % m/m and these are obtained after carrying out an interlaboratory comparison exercise (ILCE) where 12

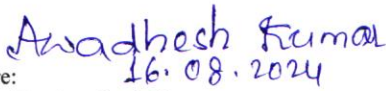
ILCE Participating laboratories

Chemistry Lab, Atomic Minerals Directorate for Exploration and Research, DAE, Bengaluru - 560 072
Chemistry Lab, Atomic Minerals Directorate for Exploration and Research, DAE, Hyderabad – 501301
Chemistry Lab, Atomic Minerals Directorate for Exploration and Research, DAE, Jaipur – 302033
Chemistry Lab, Atomic Minerals Directorate for Exploration and Research, DAE, Nagpur – 440001
Chemistry Lab, Atomic Minerals Directorate for Exploration and Research, DAE, Shillong - 973019
Control Laboratory, Nuclear Fuel Complex (NFC), DAE, Hyderabad -500062.
National Centre for Compositional Characterization of Materials, Bhabha Atomic Research Centre (NCCCM-
BARC), DAE, Hyderabad-500062
NALCO Research & Technology Centre, Bhubaneswar, Odisha - 751003.
NALCO R&D Department, Mines & Refinery Complex, Damanjodi, Koraput, Odisha - 763008.
Jawaharlal Neheru Aluminium Research and Design Centre, Amaravati Road, Wadi, Nagpur - 440023.
First Source laboratory solutions, LLP, IDA, Nacharam, Hyderabad, 500 076
SGS India Private Limited, Ambattur Industrial Estate, Chennai - 600058.

Legal disclaimer

The certified values of trace impurities 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.

Signature:


16.08.2024
Name: Dr. Awadhesh Kumar
Affiliation: Associate Director, CG, BARC
Website: www.cccm.gov.in
Contact: 022-25595330

Signature:


16.08.2024
Name: Smt. Binuta Patra
Affiliation: GGM (R&D), NRTC, NALCO
Web site: <https://nalcoindia.com/>
Contact: 0674 - 2301988