NATIONAL CENTRE FOR COMPOSITIONAL CHARACTERISATION OF MATERIALS

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NCCCM

CHEMISTRY GROUP

BHABHA ATOMIC RESEARCH CENTRE

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S. No. # INDEX Page No.

Preface

National Centre for Compositional Characterisation of Materials (NCCCM) was established in the year 1993 at Hyderabad under Chemistry Group (CG) of Bhabha Atomic Research Centre (BARC), Mumbai. Since then, NCCCM has carried out analysis of various materials of electronic, optical, semiconductor, geological, pharmaceutical and biological origin. With the experience and expertise gained during the first decade (1999-2003), NCCCM embarked for the production of Certified Reference Materials (CRMs) and got NABL accreditation under ISO 17025 & ISO 17034.

In the year 2024, NCCCM-BARC in collaboration with NALCO, Bhubaneswar produced and released the Alumina CRM (BARC-B1301). Another CRM of high strength and low alloy (HSLA) steel (DMRL-BARC-HSLA-01) certified for 11 elements is produced in collaboration with DMRL, Hyderabad and is ready for release. This CRM is an import substitute for three HSLA steel CRMs. NCCCM-BARC has started Ferrocabonatite CRM production in collaboration with AMDER, Hyderabad and homogeneity assessment was completed for 13 rare earth elements (REEs) and 6 matrix elements. The CRM is ready for release now. NCCCM-BARC has carried out mapping of coal fly ash (CFA) for Li, Ga, Ge and REEs. For the first time, NCCCM-BARC has reported the enrichment of Ge (a critical element) in CFA in the ESPs (6th and 7th field) of two NTPC plants at 47 ppm.

Novel analytical methodology was developed for multi elemental analysis in pegmatite type Li ores (α-spodumene, petalite, lepidolite). New methodologies were developed for separation, extraction and preconcentration of Sc from CFA, determination of carbide forming element Sc at trace levels in CFA, determination of Pb at ultra-trace levels in petrochemical effluents and extraction and quantification of REEs from geological materials. Besides, methodology based on *cw*-laser excited atomic fluorescence spectrometry was developed for Rb quantification in spodumene.

Ion Beam Analysis (IBA) is a modern, multi elemental, non-destructive nuclear analytical technique widely used to probe the surface and near surface regions of materials and it was employed using a diverse range of ion probes including H, He, C, Ni etc., for isotopic determination, surface analysis and modification of various materials. B-10 isotopic abundance was determined in boron carbide powder samples meant for export for a private industry. The precision of this B^{10}analysis was < 1% (RSD), which was required by the industry. In a significant advancement, we have integrated IBA with a state-of-the-art high-speed digitizer to enable prompt coincidence measurements, leading to the development of Coincidence Ion Beam Analysis (CIBA).

Service analysis of a variety of samples is carried out using instrumental techniques such as GFAAS, GD-MS, ICP-OES, IBA, XRF, XRD, AFM and SEM. Important samples analyzed are: boron carbide, spodumene, lepidolite, geological materials, semiconductor grade solvents, bacterial extracts, raw quartz, high purity quartz, high purity alumina, cerium oxide, $Ni(OH)_2$, Bi electrodes, BF_3CaF_2, $BF_3O(C_2H_5)_2$, copper tailings, lithium ferrite, zirconium carbide, W-Ta-Nb wear resistant coatings, Cr/Zr bilayer films, chromium nitride films, Pb-16Li alloy, scrap alloys etc.

We are immensely grateful to our esteemed Associate Director, Dr. A. C. Bhasikuttan for his continuous support, encouragement and guidance.

Thanks to my colleagues of NCCCM for their dedicated and collective efforts in fulfilling the activities pertaining to the mandate of the Centre.

K. Dash
(Head-NCCCM)

Annual Peer Review - 2024 of NCCCM-BARC, Chemistry Group, Hyderabad

1. A Journey over three decades of NCCCM – One day theme meeting

1.1. Material Characterisation - Quality Assurance in Analytical Measurements (MCQAAM-2024) - NCCCM theme meeting

One day theme meeting titled ***“Material Characterisation - Quality Assurance in Analytical Measurements (MCQAAM-2024)”*** was organized on 13th September, 2024 to commemorate 30 years of excellence in analytical measurements at NCCCM-BARC, Hyderabad.

Dr. Awadhesh Kumar, Associate Director, Chemistry Group (CG), BARC, Mumbai & Chairman MCQAAM-2024, welcomed the distinguished guests and invitees for this theme meeting. The vision for this project was highlighted by dwelling into the factors leading to the formation of NCCCM at Hyderabad. Dr. Awadhesh Kumar recounted the efforts involved in setting up a pilot plant at South site, BARC to demonstrate the capabilities of establishing a clean room facility to carry out analytical measurements at ultra-trace levels. The genesis of NCCCM was well accounted. Dr Awadhesh Kumar thanked Director, BARC for his support to the programs of NCCCM.

Dr. Komal Kapoor, Chief Executive, NFC, Chief Guest of the meeting, recapitulated the efforts of Shri K. Balarama Murthy, the then Chief Executive, NFC, and Dr. S. Gangadharan, Project Director in envisioning and establishing this National Centre in the year 1993. He mentioned about the various collaborative programs between NCCCM-BARC and Control Lab. NFC and applauded the efforts of scientists of NCCCM-BARC in carrying out important compositional analyses in materials of technological importance. He further emphasized the need for Certified Reference Materials (CRMs) production which facilitates the validation of analytical measurements and expressed his pleasure with the quantum of work carried out at NCCCM in this field.

Dr. R. Balamuralikrishnan, Director, DMRL, Hyderabad, Guest of Honour of this theme meeting, stressed the importance of material characterization in the development of materials from the perspective of a metallurgist. He welcomed the creation of a national institute completely dedicated to analytical measurements in trace and ultra-trace levels, such as NCCCM-BARC, by Department of Atomic Energy. He exhorted to have an active collaboration between NCCCM and DMRL in the production of certified reference materials (CRMs) and compositional analysis of strategic materials.

Shri K. Srinivas, Director, ESG, BARC, Mumbai, Guest of Honour of this theme meeting, remembered his nostalgic experiences as a young civil engineer for this project. He described the efforts of the engineers and complexities involved in building the first non-metallic clean lab in India at NCCCM-BARC.

Dr. K. Dash, Head, NCCCM-BARC, enumerated few important activities carried out since past three decades. He highlighted the importance of production of CRMs in analytical measurements and described in detail about the ongoing works in this regard.

Eminent Scientists from CG, RCIG, BARC and other DAE institutes like IGCAR, AMDER delivered invited talks encompassing various topics including materials development using high energy radiation, chemical sensors, quality assurance in materials characterization, wastewater treatment, application of radiation & photochemistry, analytical characterization of geological materials, quantification of low Z elements by nuclear methods etc.

A souvenir, encompassing important activities of NCCCM-BARC in the past three decades, was released on this occasion along with inauguration of new name plaque of NCCCM-BARC at Hyderabad.

Delegates of one day theme meeting on Material Characterisation - Quality Assurance in Analytical Measurements (MCQAAM-2024) released a souvenir at NCCCM-BARC.Left to right, Dr. Awadhesh Kumar, Dr. R. Balamuralikrishnan, Dr. Komal Kapoor, Shri K. Srinivas and Dr. K. Dash.

Inauguration of a new name plaque of NCCCM-BARC during the MCQAAM-2024 theme meeting. Left to right, Dr. R. Balamuralikrishnan, Dr. Komal Kapoor, Dr. Awadhesh Kumar and Shri K. Srinivas.

Participants of one day theme meeting of MCQAAM-2024 pose for a group photo during the inauguration of a new name plaque of NCCCM-BARC.

2. Production of certified reference materials (CRMs)

2.1. Production of alumina CRM in collaboration with NALCO Research and Technology Centre (NRTC), Bhubaneswar, Odisha

The project of production of alumina CRM was jointly carried out by National Centre for Compositional Characterisation of Materials (NCCCM) of Bhabha Atomic Research Centre (BARC) and NRTC - NALCO. A method was developed and validated using acid assisted microwave digestion and subsequent quantitation (Na_2O, MgO, CaO, TiO_2, V_2O_5, MnO, Fe_2O_3, ZnO and Ga_2O_3) by ICP-OES and ICP-MS. The alumina candidate CRM was obtained from NRTC, which was processed and 154 bottles each containing (~ 85 g) of material was produced. Within and between bottle homogeneity assessment for this batch was completed during 2023.

Further, in continuation with this project, inter-laboratory comparison exercise (ILCE), value assignment and release of alumina certified reference material (CRM) were carried out during this reported period (2024). ILCE for candidate alumina CRM for certification of 10 property values was conducted. After initial communication a total of 14 laboratories of various government and private sectors agreed to participate in the ILCE. After obtaining consent from the participating laboratories, samples and quality control material (QCM) along with standard operating procedure (SOP) of validated method has been sent to all the laboratories. The ILCE results for candidate alumina CRM were compiled and robust statistics was applied on the data remaining in pool after outlier tests for the assignment of property values.

Alumina CRM (BARC - B1301) was certified for six metallic impurities (Na_2O, CaO, Fe_2O_3, TiO_2, Ga_2O_3 and V_2O_5) are given as follows. Further, five indicative values (MgO, ZnO, MnO, LOI and surface area) were also provided. This CRM was produced as per ISO 17034:2016 and ISO guide 35:2017. The certified values are traceable to SI units. This alumina CRM (BARC - B1301) was released on 16th August, 2024 at NRTC-NALCO, Bhubaneswar, by BARC and NALCO authorities.

Certified property values and associated expanded uncertainties of metallic impurities in Alumina CRM (BARC - B1301) produced by NCCCM-BARC in collaboration with NRTC.

Impurities	Certified Values Mass Fraction (% m/m)	Expanded uncertainty (% 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

Release of Alumina CRM (BARC - B1301) on 16th August, 2024 at NRTC, NALCO, Bhubaneswar by BARC and NALCO authorities.

2.2. Work progress on production of high strength and low alloy steel CRM in collaboration with DMRL

High strength low alloy (HSLA) steel is used in naval ships and aircraft carriers. It provides improved mechanical properties and greater atmospheric corrosion resistance than traditional steel. The composition of this steel was developed by DMRL and subsequently produced by Indian industries. The quality control of this material requires three different imported CRMs. So, NCCCM-BARC and DMRL-DRDO have jointly taken up this project to produce a single CRM which can be an import substitute. The candidate CRM material of HSLA steel was provided by DMRL in the form of a steel plate (~ 20 kg). At NCCCM-BARC the physical processing of this plate was carried out and two forms (chip and disk) of candidate CRM materials were produced. A method was developed and validated for the analysis of Cr, Cu, Mn, Ni, Nb, Ti, Si, V, and P in HSLA using ICP-OES. In continuation with the works carried out during 2023, following progress has been made for the production of HSLA steel CRM during 2024.

2.2.1. Homogeneity assessment within and between bottles of high strength and low alloy (HSLA) steel candidate CRM material

A total of 86 bottles each containing ~ 90 g of candidate HSLA steel CRM were prepared for the certification of Cr, Cu, Mn, Ni, Nb, Ti, Si, V, C, S and P. Within and between homogeneity assessment was carried out for this batch by selecting 10 bottles at a fixed interval of 9. Three aliquots were taken from each bottle and digested as per validated method. The determinations of analytes were carried out using ICP-OES after suitable dilution at NCCCM-BARC and DMRL facilities. Statistical evaluation of the results was carried out for homogeneity assessment using one-way ANOVA test for the analytes including Cr, Cu, Mn, Ni, Nb, Ti, Si and V, which were previously analysed by ICP-OES. $F_{calculated}$ was found to be less than $F_{critical}$ for all the 11 property values (Cr, Cu, Mn, Ni, Nb, Ti, Si, V, C, S and P). Thus, the homogeneity of candidate HSLA was established within and between units with respect to all the tested property values. Within and between homogeneity assessment for C, S and P was carried out by selecting 10 bottles at a fixed interval of 9. Three aliquots were taken from each bottle and analyzed for these property values. Analysis of C and S was carried out using C-S analyzer. P was analyzed by an in–house validated spectrophotometric method.

2.2.2. Inter-laboratory comparison exercise and value assignment

A total of 21 laboratories from government, public and private sector were contacted for inter-laboratory comparison exercise (ILCE) of HSLA steel. Participating laboratories were instructed to use either the ICP-OES based method, which was validated at our laboratory or any other method available at their laboratories for the analysis of steel. A quality control material, QCM was also sent along with the candidate HSLA steel CRM to each laboratory. All the 21 laboratories submitted the report of ILCE exercise. Assignment of property values of HSLA was done using robust statistics. The certified values along with expanded uncertainty of property values of HSLA steel are given in following table. The given uncertainty of the certified value is at a confidence level 95% (Coverage factor k = 2) as per the ISO Guide 35:2017 and ISO 13528:2022. Statistical methods for use in proficiency testing by inter-laboratory comparison. Both chip and disk form of HSLA steel CRM (DMRL-BARC-HSLA-01) is given below.

Chip and disk forms of HSLA steel CRM (DMRL-BARC-HSLA-01).

Certified property values and associated expanded uncertainties of impurities in HSLA steel CRM (DMRL-BARC-HSLA-01) produced by NCCCM-BARC in collaboration with DMRL. This CRM shall be released by DAE and DRDO authorities shortly.

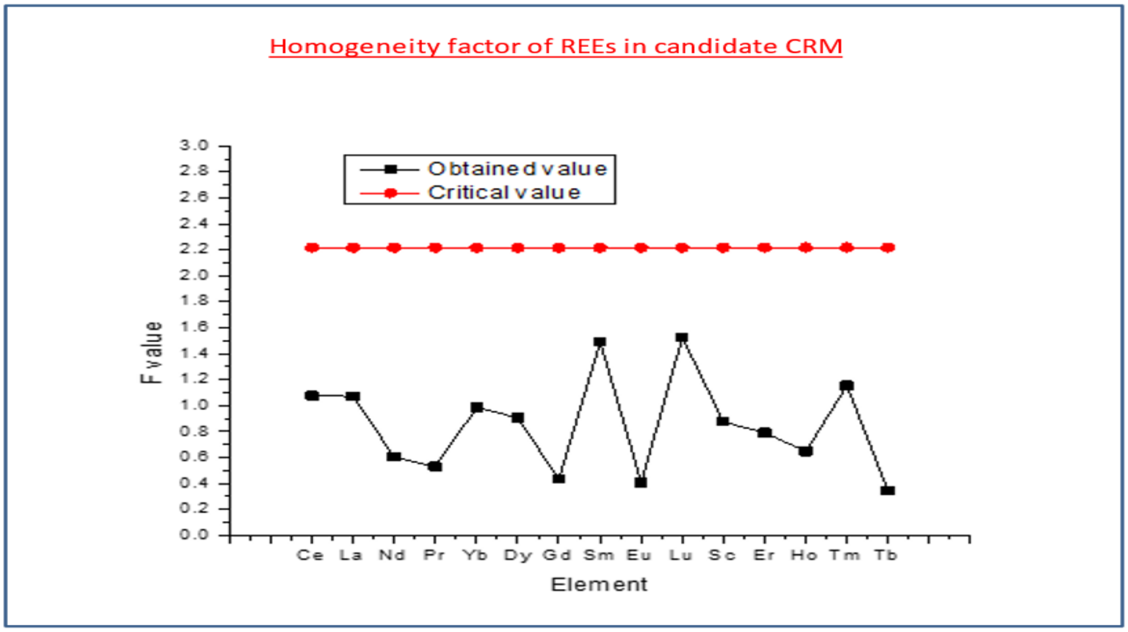
Analyte	**Certified Values ± Expanded uncertainty Mass Fraction (% m/m)**
Si	0.242 ± 0.012
Ti	0.013 ± 0.001
V	0.025 ± 0.001
Cr	0.018 ± 0.002
Mn	1.23 ± 0.03
Ni	0.78 ± 0.03
Cu	0.0081 ± 0.0005
Nb	0.050 ± 0.004
C	0.111± 0.004
P	0.0144 ± 0.0013
S	0.0059 ± 0.0008

2.3. Work progress on production of ferrocarbonatite CRM in collaboration with AMDER

2.3.1. Assessment of within and between bottle homogeneity

Rare Earth Elements (REEs) play an important role in modern electronic industries, rechargeable batteries, autocatalytic converters, super magnets, superconductors, fluorescent materials, solar panels and magnetic resonance imaging (MRI). Carbonatites associated alkaline rocks are the primary sources for REE mineralization. Ferrocarbonatite (FC) deposits found in Amba Dongar, Gujarat is well exposed above the surface in the form of hillocks spread over a 10 sq km area. Further, this FC contains high concentration of Neodymium, Praseodymium and significant amount of high-Z REEs. Indigenous recoveries of these metals are essential due to export restrictions from major REE mineral rich countries. Hence, an accurate and precise method is required for the exploration and extraction of REEs in FC. In international market only few FC reference materials are available; there is an urgent need to produce an indigenously developed Certified Reference Material, which is an import substitute. Hence, NCCCM-BARC and AMDER-Hyderabad collaborated for the indigenous production of FC-CRM for 13 REEs and 6 major elements. Various steps involved in the production of FC-CRM such as material collection, sieving, homogenization, bottling, homogeneity test of within and between the bottles, inter-laboratory comparison exercise and statistical evaluation were completed. Assignment of property values are carried out as per ISO guide 35:2017 and the FC-CRM is ready for release by DAE authorities.

During this period, within and between bottle homogeneity study has been carried out using ICP-OES and ICP-MS (NRTC-NALCO). The bulk homogenized ferrocarbonatite (FC) was divided into 100 g each in 175 bottles. Then within and between bottles homogeneity assessment was carried out by analysing three aliquots from 14 bottles. Forty-two samples were dissolved using the developed method followed by analysis using ICP-OES and ICP-MS for the determination of sixteen REEs and seven major elements (Fe, Ca, Mg, Mn, Al, Ti and P). The statistical evaluation for homogeneity for within and between bottles was carried out by using one-way ANOVA test and found out that $F_{calculated}$ values were less than the $F_{critical}$ values of all REEs and six major elements (except Ti).



$F_{calculated}$ values of studied analytes in ferrocarbonatite candidate CRM.

2.3.2. Inter-laboratory comparison exercise (ILCE)

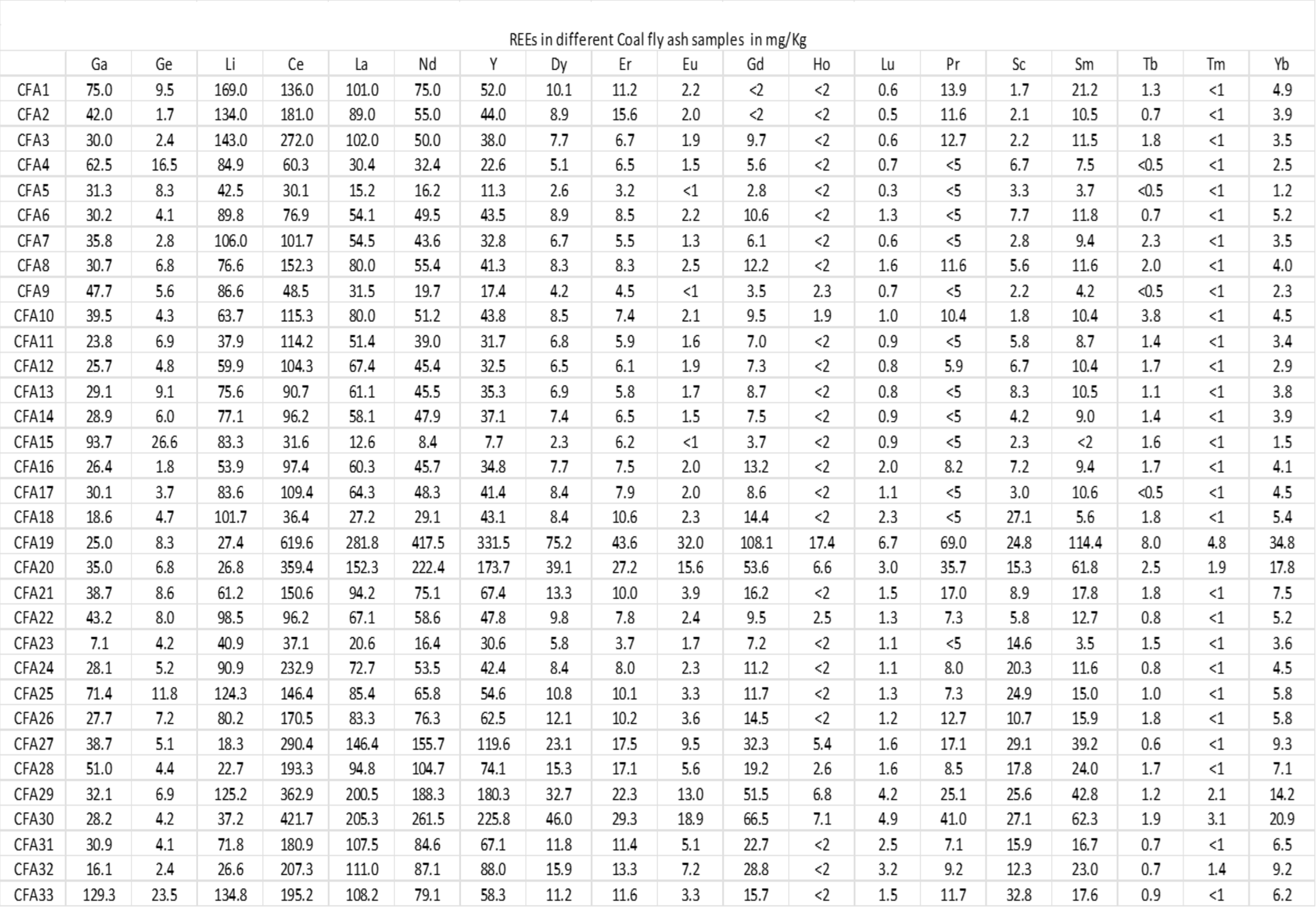
Fifteen laboratories were accepted to participate in this ILCE for the determination of 23 elements in FC. Standard operating procedure (SOP) was compiled and verified for the determination using ICP-OES or ICP-MS for all these elements. Fifteen bottles were randomly selected across 175 bottles and sent 10 g each for the participating laboratories along with quality control material (QCM).

2.4. Mapping of REEs, Ga, Ge and Li in coal fly ash (CFA) from various thermal power plants in India

2.4.1. Mapping of Indian coal fly ash (CFA) for Ga, Ge, Li and REEs

Thirty three coal fly ash samples were collected from various thermal power plants across India (6 samples from NLC collected by MPD, BARC). Mapping of Ge, Ga, Li and REEs were carried out in all 33 CFA samples and results are given in the following table. The analytical results shows that CFA obtained from NLC (lignite) contains higher REEs, but low concentration of Ga, Ge and Li. JPL-Jharli (Haryana) and Shakti Nagar, U.P (Bituminous) show reverse trend (*i.e.,* low REE and high Ga, Ge and Li). CFA obtained from Kota Super Thermal Power Station (KSTPS) contains all the interested elements at higher concentration. KSTPS receives coal from Indian mines. Six lignite-based CFA samples (CFA 27-32) were collected by Mineral Processing Division, BARC, Hyderabad.

Results of CFA samples collected from various thermal power plants in India



REEs in different Coal fly ash samples in mg/Kg

	Ga	Ge	Li	Ce	La	Nd	Y	Dy	Er	Eu	Gd	Ho	Lu	Pr	Sc	Sm	Tb	Tm	Yb
CFA1	75.0	9.5	169.0	136.0	101.0	75.0	52.0	10.1	11.2	2.2	<2	<2	0.6	13.9	1.7	21.2	1.3	<1	4.9
CFA2	42.0	1.7	134.0	181.0	89.0	55.0	44.0	8.9	15.6	2.0	<2	<2	0.5	11.6	2.1	10.5	0.7	<1	3.9
CFA3	30.0	2.4	143.0	272.0	102.0	50.0	38.0	7.7	6.7	1.9	9.7	<2	0.6	12.7	2.2	11.5	1.8	<1	3.5
CFA4	62.5	16.5	84.9	60.3	30.4	32.4	22.6	5.1	6.5	1.5	5.6	<2	0.7	<5	6.7	7.5	<0.5	<1	2.5
CFA5	31.3	8.3	42.5	30.1	15.2	16.2	11.3	2.6	3.2	<1	2.8	<2	0.3	<5	3.3	3.7	<0.5	<1	1.2
CFA6	30.2	4.1	89.8	76.9	54.1	49.5	43.5	8.9	8.5	2.2	10.6	<2	1.3	<5	7.7	11.8	0.7	<1	5.2
CFA7	35.8	2.8	106.0	101.7	54.5	43.6	32.8	6.7	5.5	1.3	6.1	<2	0.6	<5	2.8	9.4	2.3	<1	3.5
CFA8	30.7	6.8	76.6	152.3	80.0	55.4	41.3	8.3	8.3	2.5	12.2	<2	1.6	11.6	5.6	11.6	2.0	<1	4.0
CFA9	47.7	5.6	86.6	48.5	31.5	19.7	17.4	4.2	4.5	<1	3.5	2.3	0.7	<5	2.2	4.2	<0.5	<1	2.3
CFA10	39.5	4.3	63.7	115.3	80.0	51.2	43.8	8.5	7.4	2.1	9.5	1.9	1.0	10.4	1.8	10.4	3.8	<1	4.5
CFA11	23.8	6.9	37.9	114.2	51.4	39.0	31.7	6.8	5.9	1.6	7.0	<2	0.9	<5	5.8	8.7	1.4	<1	3.4
CFA12	25.7	4.8	59.9	104.3	67.4	45.4	32.5	6.5	6.1	1.9	7.3	<2	0.8	5.9	6.7	10.4	1.7	<1	2.9
CFA13	29.1	9.1	75.6	90.7	61.1	45.5	35.3	6.9	5.8	1.7	8.7	<2	0.8	<5	8.3	10.5	1.1	<1	3.8
CFA14	28.9	6.0	77.1	96.2	58.1	47.9	37.1	7.4	6.5	1.5	7.5	<2	0.9	<5	4.2	9.0	1.4	<1	3.9
CFA15	93.7	26.6	83.3	31.6	12.6	8.4	7.7	2.3	6.2	<1	3.7	<2	0.9	<5	2.3	<2	1.6	<1	1.5
CFA16	26.4	1.8	53.9	97.4	60.3	45.7	34.8	7.7	7.5	2.0	13.2	<2	2.0	8.2	7.2	9.4	1.7	<1	4.1
CFA17	30.1	3.7	83.6	109.4	64.3	48.3	41.4	8.4	7.9	2.0	8.6	<2	1.1	<5	3.0	10.6	<0.5	<1	4.5
CFA18	18.6	4.7	101.7	36.4	27.2	29.1	43.1	8.4	10.6	2.3	14.4	<2	2.3	<5	27.1	5.6	1.8	<1	5.4
CFA19	25.0	8.3	27.4	619.6	281.8	417.5	331.5	75.2	43.6	32.0	108.1	17.4	6.7	69.0	24.8	114.4	8.0	4.8	34.8
CFA20	35.0	6.8	26.8	359.4	152.3	222.4	173.7	39.1	27.2	15.6	53.6	6.6	3.0	35.7	15.3	61.8	2.5	1.9	17.8
CFA21	38.7	8.6	61.2	150.6	94.2	75.1	67.4	13.3	10.0	3.9	16.2	<2	1.5	17.0	8.9	17.8	1.8	<1	7.5
CFA22	43.2	8.0	98.5	96.2	67.1	58.6	47.8	9.8	7.8	2.4	9.5	2.5	1.3	7.3	5.8	12.7	0.8	<1	5.2
CFA23	7.1	4.2	40.9	37.1	20.6	16.4	30.6	5.8	3.7	1.7	7.2	<2	1.1	<5	14.6	3.5	1.5	<1	3.6
CFA24	28.1	5.2	90.9	232.9	72.7	53.5	42.4	8.4	8.0	2.3	11.2	<2	1.1	8.0	20.3	11.6	0.8	<1	4.5
CFA25	71.4	11.8	124.3	146.4	85.4	65.8	54.6	10.8	10.1	3.3	11.7	<2	1.3	7.3	24.9	15.0	1.0	<1	5.8
CFA26	27.7	7.2	80.2	170.5	83.3	76.3	62.5	12.1	10.2	3.6	14.5	<2	1.2	12.7	10.7	15.9	1.8	<1	5.8
CFA27	38.7	5.1	18.3	290.4	146.4	155.7	119.6	23.1	17.5	9.5	32.3	5.4	1.6	17.1	29.1	39.2	0.6	<1	9.3
CFA28	51.0	4.4	22.7	193.3	94.8	104.7	74.1	15.3	17.1	5.6	19.2	2.6	1.6	8.5	17.8	24.0	1.7	<1	7.1
CFA29	32.1	6.9	125.2	362.9	200.5	188.3	180.3	32.7	22.3	13.0	51.5	6.8	4.2	25.1	25.6	42.8	1.2	2.1	14.2
CFA30	28.2	4.2	37.2	421.7	205.3	261.5	225.8	46.0	29.3	18.9	66.5	7.1	4.9	41.0	27.1	62.3	1.9	3.1	20.9
CFA31	30.9	4.1	71.8	180.9	107.5	84.6	67.1	11.8	11.4	5.1	22.7	<2	2.5	7.1	15.9	16.7	0.7	<1	6.5
CFA32	16.1	2.4	26.6	207.3	111.0	87.1	88.0	15.9	13.3	7.2	28.8	<2	3.2	9.2	12.3	23.0	0.7	1.4	9.2
CFA33	129.3	23.5	134.8	195.2	108.2	79.1	58.3	11.2	11.6	3.3	15.7	<2	1.5	11.7	32.8	17.6	0.9	<1	6.2

2.4.2. Rapid method for the determination of Ga and Ge in coal fly ash using ICP-OES

A rapid sample digestion method has been developed for the determination of strategically important secondary resource elements (Ge and Ga) in coal fly ash (CFA) using Inductively Coupled Plasma Optical Emission Spectrometry (ICP-OES). In this developed method, a non-corrosive ammonium bi fluoride ($NH_4.HF_2$) with dilute H_2SO_4 has been utilized for the digestion of CFA on hotplate. This salt-acid mixture liberates HF from $NH_4.HF_2$ with the formation of oxidizing flux (NH_4HSO_4) for rapid digestion of glassy phases of CFA. After the addition of concentrated HCl at room temperature, diluted for required volume and measured the analytes using ICP-OES. The developed method has been applied for the analysis of variety of CFA samples. The method validation has been carried out using SRM 1633b.

2.4.3. Pre-concentration of germanium (Ge) and gallium (Ga) in coal fly ash collected from electrostatic precipitators of thermal power plants

Coal fly ash (CFA) is considered to be secondary resource for Germanium (Ge), Gallium (Ga) and Lithium (Li). Due to large volume of CFA generated in Indian thermal power plants; NCCCM-BARC has carried out the mapping of Ge, Ga and Li in mixed coal fly ash from Unit - 6 of Kota Super Thermal Power Station (KSTPS), Rajasthan. In this mapping exercise, Ge concentration is found to be in the range of 2 to 10 mg kg^{-1}, extraction of such low concentration of Ge is not economically viable. Therefore, the samples were collected from different fields of electrostatic precipitators (ESP) from KSTPS, Rajasthan; Bhadradri Thermal Power Station-3 (BTPS-3), Telangana and National Thermal Power Corporation (NTPC), Shaktinagar, UP. The results have shown significant pre-concentration of Ge (~ 6 times) in the 6th field of ESP from above mentioned thermal power plants.

Results of the Ge, Ga and Li in CFA samples collected from different fields of ESP, KSTPS, Kota.

S. N.	**Sample**	**Concentration (mg kg^{-1})**		
		Ge	Ga	Li
1	1st field	8.1	18.6	59.5
2	2nd field	18.9	29.2	63.4
3	3rd field	21.4	44.6	95.5
4	4th field	27.1	53.2	65.3
5	5th field	41.3	60.6	95.2
6	6th field	46.8	62.2	73.0

Results showing pre-concentration of Ge in CFA samples collected at different fields of ESPs from various thermal power plants.

Sample	Concentration (mean ± SD, mg kg^{-1}) (n = 3)		
	KSTPS, Rajasthan	BTPS-3, Telangana	NTPC, Shaktinagar, Uttar Pradesh
1st field	8.1 ± 0.6	6.2 ± 0.4	--
2nd field	18.9 ± 0.9	--	--
3rd field	21.4 ± 0.9	--	--
4th field	27.1 ± 1.2	--	--
5th field	41.3 ± 2.0	19.1 ± 0.8	26.0 ± 1.0
6th field	46.8 ± 2.1	32.2 ± 1.2	36.1 ± 1.4

2.5. Preparation of wheat flour reference material (RM)

In the preparation of a reference material, RM of Wheat Flour, the between to within bottle homogeneity of the 100 bottles filled with wheat flour were carried out. 10 bottles were selected randomly from the 100 bottles following systematic sampling method. Three replicates from each bottle were weighed (~ 0.5 g) and digested using a dilute HNO_3/H_2O_2 mixture, following a standard operating procedure. A certified reference material (NIST 1567b) was also digested for validation of the method. The digests were analyzed by ICP-OES for Fe and Zn. The data obtained from the 30 digested solutions were analyzed statistically using one-way ANOVA test. The F-value calculated ($F_{calculated}$) for both Fe ($F_{calculated}$ = 1.17) and Zn ($F_{calculated}$ = 0.40) were less than the critical value ($F_{critical}$ = 2.39), demonstrating the between to within bottle homogeneity for Fe and Zn in wheat flour. The concentration of Fe and Zn were found to be 51.0 ± 1.7 and 31.2 ± 0.6 mg/kg. The preparation of 100 bottles of wheat flour reference material was completed.

The certificates of various certified reference materials (CRMs) produced at NCCCM-BARC are included below.

National Centre for Compositional Characterisation 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.

The alumina powder material for trace impurities 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 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 at105°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)

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

[2] ISO 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 ±2.SD	Analytical method
MgO	% m/m	0.0011 ± 0.0003	ICP-AES
MnO	% m/m	0.0003 ± 0.0001	ICP-MS
ZnO	% m/m	0.0008 ± 0.0003	ICP-MS
Loss On Ingnition (300 - 1000°C)	% m/m	1.06 ± 0.04	Thermogravimetry
Surface area	m^2/gm	71.7 ± 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 106μ sieve. A total of 13.2 kg processed material (<106μ) 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 Na_2O, MgO, CaO, TiO_2, V_2O_5, MnO, Fe_2O_3, ZnO and Ga_2O_3 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 BET analysis respectively at NRTC Bhubaneshwar 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 measurement for homogeneity study.

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 ± 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 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

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

RC-1026

National Centre for Compositional Characterisation 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 (Al_2O_3, Fe_2O_3, SiO_2, TiO_2, Cr_2O_3, MgO, MnO, V_2O_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 Spectrometry (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[1] Mass Fraction (% m/m)**	**Expanded uncertainty[2] (% m/m)**
Al_2O_3	41.3	0.5
Fe_2O_3	29.3	0.5
SiO_2	4.12	0.23
TiO_2	2.33	0.05
Cr_2O_3	0.044	0.003
MnO	0.057	0.005
MgO	0.027	0.003
V_2O_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)

[1] ISO 13528 (2015): Statistical methods for use in proficiency testing by inter-laboratory comparison, [2]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 ± SD	Analytical method
Ga_2O_3	% m/m	0.0094 ± 0.0005	ICP-MS
ZnO	% m/m	0.008 ± 0.001	ICP-MS
CaO	% m/m	0.009 ± 0.001	ICP-AES
Extractable Alumina[1]	% m/m	34.2 ± 0.4	Titrimetry

[1] 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 106μ sieve. A total of 14.4 kg processed material (<106μ) 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_2O_3, Fe_2O_3, SiO_2, TiO_2, Cr_2O_3, MgO, MnO, V_2O_5 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 ± 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 March, 2023 and its further long term stability study would continue at regular intervals (2 years) until stock lasts. This certificate is valid till 29th February 2033, 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

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. Durga Prasad, Dr. Lori Rastogi and Dr. K. Dash. The data analysis and statistical evaluation for certification has been done by Sri A. Durga Prasad, 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-400085.
Chemistry Lab, Atomic Minerals Directorate for Exploration and Research, DAE, Bengaluru-560072.
Chemistry Lab, Atomic Minerals Directorate for Exploration and Research, DAE, Hyderabad-501301.
Chemistry Lab, Atomic Minerals Directorate for Exploration and Research, DAE, Jaipur302033.
Chemistry Lab, Atomic Minerals Directorate for Exploration and Research, DAE, Nagpur-440001.
Chemistry Lab, Atomic Minerals Directorate for Exploration and Research, DAE, New Delhi-110066.
Chemistry Lab, Atomic Minerals Directorate for Exploration and Research, DAE, Jamshedpur-973010.
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 safety (Pl. see the material safety data sheet) 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: 24/3/2023
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RC-1026

National Centre for Compositional Characterisation of Materials (NCCCM), Bhabha Atomic Research Centre (BARC) Hyderabad, India

प्रमाणित निर्देशक द्रव्य

Certified Reference Material
Major and Minor Constituents in Tea Powder
Reference Material Certificate

BARC-D3201

Certified Reference Material (CRM) of tea for major and minor constituents (K, Ca, P, Mg, Mn, Al, Fe, Ba, Zn, Cu, Sr, Pb, As, Cd, and Hg) 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 in the routine analysis of tea powder. One bottle of this CRM contains ~ 20 g of the tea powder in an amber colour glass bottle.

The tea 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. The results are referred to the dry material (1g) corrected for moisture at 85°C for 5 h. Analytical techniques used for the determination of major and minor constituents in tea powder by the participant laboratories include inductively coupled plasma atomic emission spectrometry (ICP-AES), graphite furnace atomic absorption spectrometry (GFAAS), include inductively coupled plasma mass spectrometry (ICP-MS), atomic fluorescence spectrometry (AFS), and Hydride Generation Atomic Absorption Spectrophotometry **(**HGAAS). This tea certified reference material (CRM) was produced in accordance with the ISO GUIDE 34: 2016 and ISO/IEC 17025:2017. Assigned property values were established according to ISO Guide 35:2017 guidelines.

Analyte	Certified Values[1]	Expanded uncertainty[2] (k =2)	Units
Ca	0.458	0.025	% m/m
K	1.99	0.139	% m/m
P	0.303	0.031	% m/m
Mg	0.225	0.014	% m/m
Mn	1150	75.0	mg kg^{-1}
Al	1033	94.0	mg kg^{-1}
Fe	431	29.5	mg kg^{-1}
Ba	44.7	2.3	mg kg^{-1}
Zn	25.5	1.1	mg kg^{-1}
Cu	19.1	0.79	mg kg^{-1}
Sr	15.3	1.27	mg kg^{-1}
Pb	3.45	0.31	mg kg^{-1}
Cd	1.27	0.11	mg kg^{-1}
Hg	0.60	0.084	mg kg^{-1}
As*	1.45	0.36	mg kg^{-1}

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

[1] ISO 13528 (2015): Statistical methods for use in proficiency testing by inter-laboratory comparison, [2]ISO 35: 2017 guidelines, * Indicative value

Origin and preparation of the tea CRM material

Tea granules used for the preparation of candidate tea CRM were procured from local market. A total of 3.6 kg processed material was obtained and packed in 146 glass bottles each containing ~20 g of material.

Homogeneity study

Minimum sample size used for analytical homogeneity is ~500 mg for all the analytes. Bulk homogeneity was established prior to packing the tea powder into 146 bottles. Within and between bottles homogeneity tests for K, Ca, P, Mg, Mn, Al, Fe, Ba, Zn, Cu, Sr, Pb, As, Cd, and Hg were carried out at National Centre for Compositional Characterisation of Materials (BARC-NCCCM), Hyderabad using a validated method based on microwave digestion followed by ICP-AES, GFAAS and AFS measurement. Homogeneity tests were carried out as per ISO 17034:2016 and ISO Guide 35:2017 and the inhomogeneity as evaluated using one way ANOVA is not significant but the same is incorporated into the uncertainty of the certified values.

Instructions for Handling & storage

This CRM should be stored at 4°C temperature. 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 well before taking the CRM aliquot sample.

Expiration of Certification

Tea powder sample is stable since last twelve months. The material is expected to be stable for next twenty-four months when stored at 4 °C and its further long term stability study would be checked every six months until stock lasts. 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

Coordination for this CRM production was carried out by Dr. K. Dash, Dr. Lori Rastogi and Shri A. Durga Prasad of NCCCM-BARC. The processing (grinding, sieving and homogenization) of CRM material was carried out at NCCCM-BARC by Sri A. Durga Prasad, Dr. Lori Rastogi and Dr K. Dash. 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-D3201 certified reference material are the mass fractions of specified major and trace elements, expressed in % m/m and mg kg^{-1} respectively. These are obtained 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.

ILCE Participating laboratories

1. National Centre for Compositional Characterisation of Materials, Bhabha Atomic Research Centre, ECIL-post Hyderabad - 500 062
2. Analytical Chemistry Division Bhabha Atomic Research Centre Trombay, Mumbai – 400 085
3. Centre for Materials for Electronics Technology (C - MET) IDA Phase - III, HCL (Post), Hyderabad - 500051
4. Bureau Veritas (India) Pvt. Ltd. F-2, Phase III, Thiru Vi Ka Industrial Estate Chennai – 600032
5. Analytical Chemistry Group Defence Metallurgical Research Laboratory Kanchanbagh, Hyderabad-500058
6. Export Inspection Agency Ministry of commerce and Industry, Kolkata – 700107
7. Export Inspection agency –Kochi Laboratory Ministry of Commerce and Industry, Kochi – 682036
8. FirstSource Laboratory Solutions LLP, IDA, Nacharam, Hyderabad, 500 076
9. Indian Institute of Food Processing Technology Ministry of Food Processing Industries (MoFPI), Thanjvur – 613005
10. National Commodities Management services Limited, IDA –Uppal, Hyderabad 500 039
11. SGS India Private Limited, Ambattur Industrial Estate, Chennai - 600058

Analytical Method

After shaking the bottle well about 500 mg sample aliquot is recommended to be taken for acid digestion. The recommended sample processing is microwave assisted acid digestion method by conc. nitric acid (2 mL, HNO_3), hydrogen peroxide (3 mL, 30 %, v/v) and 5ml DI water (the microwave digestion step includes a hold time of 30 min at 220 - 230°C). Quantification was carried out using external calibration. Analysis of samples should be done after suitable dilutions. The moisture content should be determined by drying a separate portion of sample at 85°C for 5 hr.

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 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.

Validity: 30th April, 2026

Awadhesh Kumar
08. 10. 2024

Signature:
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RC-1026

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 बी११०१**

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)
[1] ISO 35: 2017 guidelines, [2] ISO 13528 (2015): Statistical methods for use in proficiency testing by inter-laboratory comparison.

The given uncertainty of the certified value is at a confidence level 95% (coverage factor k=2)
[1]ISO 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 thorough 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 ~105°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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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[1] (mg kg^{-1})	Expanded uncertainty[2] (mg kg^{-1})
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)

[1] ISO 35: 2006 guidelines

[2] 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 µm 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 handing 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 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 @ ~110 °C for two hours (negligible moisture content, ~< 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 (HNO_3) and thereafter, the silicon matrix was evaporated as 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.

Date of Issue: 01-02.2016

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Bhabha Atomic Research Centre (BARC), Hyderabad-500062

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भारतीय निर्देशक द्रव्य

Indian Reference Material

BND 4101.01

Trace Elements in High Purity Quartz

Date: 04.03.2024

Addendum-2

Extension of Validity:

Based on the stability tests carried out by National Centre for Compositional Characterisation of Materials (NCCCM – BARC), the validity of this certificate has been extended up to 30th April 2033.

Authorized Signatory

Dr. K. Dash
Head, NCCCM-BARC
Hyderabad - 500 062
India

डॉ. के. दाश / Dr. K. Dash
प्रधान / Head
एनसीसीसीएम, भा.प.अ. केंद्र / NCCCM, BARC,
भारत सरकार / Government of India,
हैदराबाद / Hyderabad-500062.

3. Development of analytical methodologies for the analysis of new samples

3.1. Compositional characterization of CaF_2:BF_3 and $BF_3O(C_2H_5)_2$

CaF_2:BF_3 samples were received from ECIL, Hyderabad. Nuclear grade BF_3 is produced by thermal decomposition of calcium fluoride and boron tri fluoride complex (CaF_2:BF_3). BF_3 gas produced by this method is used in proportional counters for neutron detection. Recent consignment of this complex was found to give low yield of BF_3 and unknown impurities, which are not condensed even at liquid nitrogen temperature. NCCCM-BARC analyzed both old and new CaF_2:BF_3 complexes. The samples were analyzed for total boron (B), total calcium (Ca), trace elements (Mg and Si), fluoride and SO_2 concentration. CaF_2:BF_3 and $BF_3O(C_2H_5)_2$ samples were analyzed for Heavy Water Plant (HWP), Talcher.

3.2. Determination of SO_2 in CaF_2:BF_3 by UV-visible spectrophotometric methods

The electronegative gas impurities such as SO_2 strongly affect the efficiency of BF_3 in proportional counter. The impurities produce spurious pulses due to the formation of negative ions near to the surface of the electrode. Around 0.25 g CaF_2:BF_3 sample was treated with water and un-dissolved matter (CaF_2) was filtered out. The absorbance of the sample solution in dilute H_2SO_4 medium was measured at 275 nm using UV-visible spectrophotometer. The developed method was cross validated using rosaniline method.

Rosaniline method: Na-tetrachloromercuriate, p-rosaniline and formaldehyde were added into the extracted sample solution and the absorbance was measured at 560 nm using spectrophotometer. Analysis results of these samples are shown in following tables.

Determination of B, F^-, BF_3 and SO_2 in $BF_3O(C_2H_5)_2$ samples from HWP, Talcher

Sample code	Concentration (mean ± SD, % m/m) (n = 5)			
	B*	F^{-**}	$BF_3^{\#}$	SO_2**
Old $BF_3O(C_2H_5)_2$	8.3 ± 0.3	43 ± 2	48 ± 2	<0.05
New $BF_3O(C_2H_5)_2$	8.4 ± 0.3	43 ± 2	48 ± 2	<0.05

*- ICP-OES, $^{\#}$ - Titration, **- Spectrophotometer.

Determination of B, BF_3, SO_2, Ca, Mg and Si in various CaF_2:BF_3 samples from HWP, Talcher

Sample code	**Concentration (mean ± SD, % m/m) (n = 5)**					
	B*	**BF_3#**	**SO_2****	**Ca***	**Mg***	**Si***
1	3.8 ± 0.2	18 ± 1	<0.05	37 ± 1	1.1 ± 0.1	0.62 ± 0.03
2	3.5 ± 0.2	15 ± 1	<0.05	38 ± .1	0.06 ± 0.01	0.14 ± 0.02
3	4.4 ± 0.2	20 ± 1	<0.05	39 ± .1	0.13 ± 0.01	0.11 ± 0.01

*- ICP-OES, # - Titration, **- Spectrophotometer.

3.3. A simplified method for the determination of total BF_3 in CaF_2:BF_3 samples by its extraction into organic Lewis base

The precise and accurate determination of BF_3 in the CaF_2:BF_3 complex is important as this data gives an idea about the exact amount of gas which will be liberated from the complex. Hence, a simple procedure is developed for the determination of BF_3 in CaF_2:BF_3 complex. Where BF_3 was extracted using diethyl ether and quantified by titrimetry. The developed method was found to give precise and accurate results on real samples. Further, results also suggested that there was no significant difference in the BF_3 concentration obtained by developed method as compared with water leach method. Moreover, the amount of Si (which needs to be corrected for final calculations) is not extracted in developed method which is advantageous. The following results shows that the concentration of total BF_3 was significantly low in new sample as compared to old sample, this could be possible reason for lower recovery of BF_3 gas.

Determination of BF_3 and leachable Si in various CaF_2:BF_3 samples from ECIL, Hyderabad.

Sample code	**BF_3# (water leach) (% m/m ± SD)**	**BF_3# (developed method) (% m/m ± SD)**	**% Si* in sample**	**% Si* extracted**
Old CaF_2:BF_3	31 ± 2	31.5 ± 0.5	0.23	< 0.01
New CaF_2:BF_3	17 ± 1	18.2 ± 0.5	0.38	<0.01

*- ICP-OES, # - Titration.

3.4. Determination of Ni, Co and trace impurities (Zn, Fe, Al, Cd, Mg and Mn) in nickel hydroxide from HBL Power systems, Hyderabad

The sample was dissolved using a mixture of H_2SO_4:$HClO_4$ in the presence of vanadium catalyst for decomposition of graphite present in the sample. Then the sample solution is measured for major and trace elements using ICP-OES and FAAS.

3.5. Development of a hotplate digestion method using a novel in-house PTFE digestor for the rapid and reliable determination of technology-critical elements in granite samples by ICP-OES

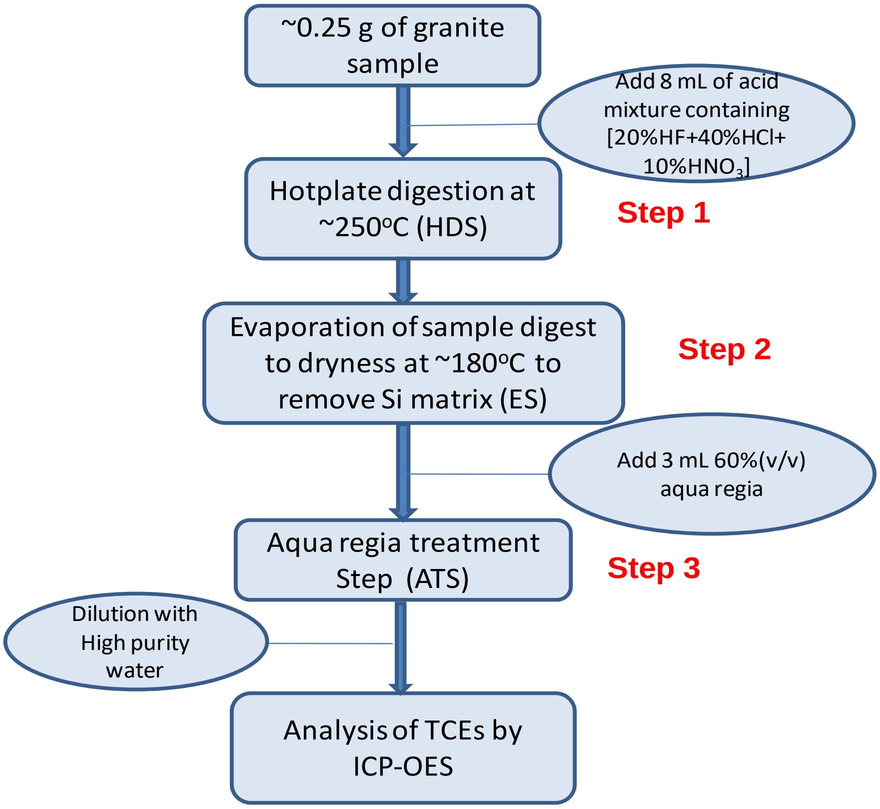
The technology-critical elements (TCEs) are vital for economic prosperity and national security of any country. The investigation of resources of TCEs such as rare earth elements (REEs) and high field strength elements (HFSEs) such as Ta, Ti, Hf, Zr and Th are very important due to their varied high-tech applications which include electronics, renewable energy systems and telecommunications. Due to the limited availability of TCEs, establishment of the new reserves of these precious metals through mineral exploration studies is growing concern across the world. Granite rocks are one of the potential sources of these rare metals. In this context, large number of granite samples need to be processed to get the reliable data for these rare metals for their potential viability to establish the base of critical raw materials. To meet this objective, a simple, rapid and reliable sample preparation method protocol is very much required for routine analysis. Sample preparation step is still regarded as a difficult step during elemental analysis, especially for complex geological matrices (e.g. granite) containing acid-resistant refractory minerals such as zircon, spinel, rutile etc.

Hence in the present work a new and high-performance PTFE digestor was designed and fabricated in-house for the total dissolution of granite samples using dilute acid mixture comprising HF, HCl, and HNO_3, by heating on a hotplate for the subsequent analysis of TCEs by ICP-OES. The cost-effective PTFE digestor is suitable for direct hotplate heating without any external jacket for the rapid and efficient digestion of granite matrices. The whole procedure comprises three simple analytical steps involving hotplate digestion aiming to dissolve the granite matrix, evaporation of silica matrix and finally aqua regia treatment for producing highly soluble chloride/nitrate complexes to obtain clear and particle-free digests. Initially, the granite sample (~ 0.25 g) was placed in the PTFE digestor and added 8 mL (v/v) of 20%HF + 40%HCl + 10%HNO_3 acid mixture. After closing, the digestor was directly heated on a hotplate at ~ 250^{o}C for about 2 h (hotplate digestion step (HDS)). Subsequently, the sample mixture was transferred to a graphite-bottom Teflon beaker for evaporation step (ES) at ~ 180^{o}C to remove silica matrix followed by aqua regia treatment step (ATS) to the sample residue for the transformation of highly insoluble meta-fluoro complexes in to soluble chloro-complexes. After ATS, the sample residue was reconstituted in 3 mL of 60% (v/v) aqua regia. The final sample digests were very clear and stable with no apparently visible fluoride precipitates indicating complete decomposition of different phases present in the granite matrix. The clear sample digests were analysed for REEs (La, Ce, Pr, Nd, Sm, Eu, Gd, Tb, Dy, Ho, Er, Yb, Sc and Y), high field strength elements HFSEs (Ta, Ti, Zr, Nb, Hf and Th) by ICP-OES.

Average recoveries obtained with the developed sample digestion method were found to be in the range of 94-107% with RSD values <10% for most the elements studied here. The trueness of the method was demonstrated through the analysis of granite based reference materials (NCSDC-73376, SARM-1 and JG-2). Various novelty aspects are involved in the present work which include (a) utilization of in-house PTFE digestor (b) hotplate heating with dilute acid mixtures (c) complete elimination of metal-fluoride precipitates (d) no need of expensive commercial bomb digestion systems and (e) total dissolution of granite sample for the determination of elements of highly technological relevance in ~ 3 h which is much faster than what is achievable using prevailing conventional methods.

(i) **(ii)** **(iii)**

(i) Top-view of the three different parts of PTFE digestor (container, lid and screw cap), (ii) PTFE vessel after closing with lid and (iii) Total view of the PTFE digestor after closing with both lid and screw cap.



Schematic diagram of the proposed hotplate digestion method utilizing the new PTFE digestor for the total dissolution of granite samples.

3.6. Development of a novel analytical method utilizing in-house PTFE digestor using NH_4HF_2 and dilute HCl for total dissolution of pegmatite-type lithium ores (α-spodumene, petalite and lepidolite): A simple scheme for the accurate multi-elemental analysis by ICP-OES

Lithium has a range of attributes such as high energy density by weight and high electrochemical potential which makes it useful for many high-tech applications. In recent years, lithium has been recognized as one of the technology-critical elements (TCEs) mainly because of its application in energy storage devices in modern technology. The base concentration levels of critical raw materials are required to be established during the mineral exploration studies. During this process, a large number of geo-samples need to be analyzed for the assessment of economic viability of these rare metals. Establishing an analytical method for the complete dissolution of the ore sample is crucial to determine rare metals to identify potential area to explore and establish the reserves of these metals. Among the pegmatite-type lithium minerals (spodumene, petalite and lepidolite), spodumene is the most widely processed lithium-bearing mineral across the globe. Spodumene exists naturally as α-spodumene possessing a monoclinic crystal structure, which has relatively high hardness and density, limiting its reactivity with acids or bases.

Hence a simple, robust and green analytical methodology, utilizing an in-house PTFE digestor, suitable for the total digestion of all the three pegmatite-type lithium ore samples (spodumene, petalite and lepidolite) was developed for the accurate determination of rare metals and other elements by ICP-OES. This procedure involve three simple analytical steps comprising digestion using a reagent mixture of NH_4HF_2 and dilute HCl followed by evaporation process to remove the silica matrix and also unreacted HF. The remaining sample residue was further treated with dilute aqua regia to convert insoluble metal-fluorides to soluble forms, which facilitate total dissolution of the test ore sample. All the solid residues obtained at each step of sample preparation were characterized by XRD and EDS analysis. The obtained clear sample digests were subsequently analyzed for the quantitative determination of rare metals (Li, Nb, Ti, Rb, Cs and Be) and other co-existing elements (Fe, Al, Mn, Na, K, Ca and Mg) by ICP-OES. In all the cases, stable, clear and particle-free sample digests were obtained with average recoveries in the range of 96-108% indicating the robustness of the method. The relative standard deviation (%RSD) values were calculated by performing five replicate measurements under optimum conditions on the same day and the obtained RSD results were in the range of 2-8%. A versatile total dissolution procedure involving three simple analytical steps was thus developed here as an alternative to the existing cumbersome digestion methods. The methodology was validated through the analysis of spodumene NBS 181, petalite NBS 182 and lepidolite NBS 183 certified reference materials. The developed method was subsequently applied to multiple ore samples of different chemical composition.

3.7. Preparation of metal functionalised polymer composites as suitable bio-sorbents for the selective separation of arsenic and phosphate in groundwater

Two different metal-functionalised polymer composites viz., La-chitosan and Ce-chitosan were prepared as suitable bio-sorbents to study the selective separation of arsenic species [As(III) and As(V)] and phosphate. The above adsorbents were prepared using the co-precipitation method. Typically, 2 g of chitosan powders were added into a 100 mL of 2% (v/v) acetic acid aqueous solution and continuously stirred for 30 min to ensure the chitosan powders have been dissolved completely. Meanwhile, metal salt solution was prepared in HCl medium by weighing known amounts of lanthanum chloride and cerium sulphate separately. The pH of the metal standard solution was adjusted to 3.5 using 1 M NaOH. Then, the obtained metal chloride solution was added to the chitosan solution drop by drop and stirred (150 rpm) for 1.5 h at room temperature to obtain a transparent viscous solution. Afterwards, the solution pH was adjusted to nearly neutral using NaOH (2.0 M) and added 10 mL of 5%

glutaraldehyde aqueous solutions as cross-linking agents and stirred intensively for 3 h until the solution gradually became viscous and resembled jelly. Finally, the metal-chitosan composites were precipitated by drop-wise addition of NaOH (2.0 M) for another 1 h, the composites were washed with ultrapure water to remove residual cross-linking agents on the surface and then dried in hot air oven at 40°C for 24 h. The yield of the composite materials was about 4 g each. The two composite materials thus prepared will be used to investigate their sorption behaviour for the selective separation of arsenic species and phosphate from groundwater.

3.8. Development of a new method for the quantification of scandium at trace and ultra-trace levels in coal fly ash and red mud by GFAAS using ammonium thiocyanate modifier

Scandium, Sc finds many applications such as Al-Sc alloy used in the aviation industry, high-end bicycle frames, and baseball bats. The radioactive isotope Scandium-46 is used as a tracer in oil refining. Currently available Sc is mainly produced as a byproduct of other processes. The separation, extraction, and pre-concentration of scandium (Sc) from industrial waste products such as coal fly ash and red mud are critical steps in recovering this valuable element. Red mud, a byproduct of the Bayer process, is rich in iron oxides, alumina, titanium dioxide, trace REEs and also scandium. A new analytical method was developed for the determination of scandium at trace and ultra-trace levels in coal fly ash (CFA) and red mud by graphite furnace atomic absorption spectrometry (GFAAS). The GFAAS detection limit for Sc was often too high due to Sc peak tailing problem, likely caused by carbide formation. This issue was resolved, and sensitivity was enhanced, by using permanent, conventional and sulfate-based modifiers and optimizing operational parameters. Maximum sensitivity and optimal peak shape were achieved using a mixed solution of 250 µg each of Ti and Ir, with 1% ammonium thiocyanate as an *in-situ* modifier. Sensitivity was further enhanced by an *in-situ* furnace pre-concentration by multiple injection mode, yielding a detection limit of 0.2 ng/mL and a characteristic mass of 11 pg. The method was successfully applied to NIST SRM 1633b coal fly ash, red mud and other CFA samples collected from various thermal power plants.

3.9. Studies on the development of a method for the separation, extraction, and pre-concentration of scandium from coal fly ash

As the recovering of this valuable element from industrial waste products such as CFA and red mud was very useful, a novel extraction and pre-concentration method utilizing room-temperature-synthesized molecular solvents synthesized using cetyltrimethylammonium bromide (CTAB) and 1-octanol was developed for the efficient extraction and matrix separation of scandium. The leaching procedure employed a mixture of acids to leach out Sc from coal fly ash. The leaching efficiency for Sc was improved with optimization of peroxide and HCl. After leaching with the acids, the extraction efficiency of Sc from leachates was studied using the synthesized molecular solvent and found to be quantitative. Stirring time was optimized to 1 h. Optimization of extractant (molecular solvent) was carried out and the extraction efficiency was found to be 85-90%.

3.10. Development of a method for the determination of lead at ppb levels in petrochemical industrial effluents using room temperature molecular solvents

A sensitive and rapid deep eutectic solvent (DES) method has been developed for the extraction, pre-concentration and determination of ultra-trace levels of lead (Pb) in petrochemical industrial effluents, contributing to effective environmental pollution monitoring. Deep eutectic solvents, known for their adjustable polarity, low melting points, and excellent solubility for both organic and inorganic compounds, enhance the efficiency of Pb extraction compared to their individual components. In this study, a hydrophobic DES synthesized at room temperature using 1-octanol and CTAB is introduced for the first time for Pb extraction. This DES demonstrates superior efficiency over individual CTAB and 1-octanol, enabling rapid phase separation without the need for centrifugation. The addition of an electrolyte further improves extraction efficiency. Pb determination is carried out using a GFAAS, ensuring high precision and sensitivity. The extraction parameters are optimized, yielding a pre-concentration factor of 25, a detection limit of 0.02 µg/L, and an analytical precision of 2-6%. The accuracy of the method is validated using certified reference materials (CRMs), including BCR-609 and BCR-610 groundwater, as well as BCR-715 industrial effluent wastewater. Since CRMs for petrochemical effluents are unavailable, spike recovery tests are performed on real samples collected from various locations of petrochemical industry outlets, with results closely matching certified and spiked values. This method is highly selective and sensitive for Pb, effectively functioning under highly acidic conditions of petrochemical effluents. By facilitating the selective extraction of Pb into a DSE, this approach offers a sustainable and reliable solution for environmental pollution monitoring, particularly for ultra-trace detection of heavy metals in complex industrial waste matrices.

3.11. Development of an analytical method for the extraction of rare earth elements from aqueous solutions and geological matrices

A novel graphene oxide (GO) mediated dispersive micro solid phase extraction method (Dµ-SPE) was developed for the extraction of rare earth elements (REEs) from seawater and leachates of coal fly-ash and determined using ICP-OES. This method involved the quantitative sorption of REE from the samples onto the negatively charged surface of GO through electrostatic interactions. The extraction was carried out in the pH range of 7-9, where GO acquires negative surface charge due de-protonation of surface functional groups such as –COOH. The increased number of reactive sites at higher pH for metal sorption improved its efficiency. The challenges posed in the separation of REE sorbed GO (REE-GO) were addressed by charge neutralization using an electrolyte, NaCl. The REE-GO was centrifuged and REEs were ultrasonically back extracted into dilute nitric acid for their determination using ICP-OES. At optimized conditions, the limits of detection for REE were in the range of 10-50 ppb with relative standard deviation of 5%-8%. The method was successfully applied for the extraction of trace REE in seawater matrices and leachates of different CFA samples. The sample preparation procedure with the GO nanomaterial was found to be simple and fast with good extraction recoveries.

3.12. Studies on the development of method for Li extraction from spent Li ion batteries (LiBs)

With the surge in electronics and EVs, recovering lithium from spent LIBs is vital for sustainability and resource conservation. In this study, a single-step ultrasonic-assisted leaching method was developed to extract lithium using eco-friendly organic acids. Discharged mobile phone batteries were dismantled, and cathode/anode materials were separated for treatment. Leaching was carried out using acids like H_2SO_4, HNO_3, H_2O_2, $HClO_4$, and V_2O_3 over 70-180 °C. Elemental analysis showed 2% Li in the anode and 5.9% in the cathode, with Co at 50% and trace metals mostly < 10 ppm. Green leaching agents such as citric, ascorbic, and oxalic acids were tested alone and in combinations, with or without oxidizers like H_2O_2, HNO_3, and HCl. Citric acid-based systems showed promising performance in extracting Li. Optimization led to a lithium leaching efficiency of up to 70%. This method offers a simplified and greener alternative for lithium recovery. It also supports circular economy goals and e-waste management.

3.13. Studies on the quantification of germanium in coal fly ash acid digests using GFAAS

Determination of Ge at ultra-trace levels by GFAAS is difficult because germanium's high volatility and presence of chlorides and sulfates cause loss of Ge during pyrolysis and thereby reducing its sensitivity and accuracy. To control these losses, various liquid modifiers (Pd, Pt, Zr, Ir) and permanent coatings of Zr and Ir were tested to optimize temperature program. Best sensitivity was achieved with 150 µg Zr coating and 10 µL 0.1% Pd as a modifier. Calibration was found to be linear for the range 30-300 ppb and Ge in the acid digests was quantified by GFAAS.

3.14. Determination of ^{6}Li/^{7}Li isotopic ratio using Zeeman splitting based graphite furnace atomic absorption spectrometry (GFAAS)

Lithium isotope ratio (^{6}Li/^{7}Li) analysis is crucial in nuclear science, geochemistry, and environmental studies. This study explores the feasibility of using Zeeman effect-based GFAAS for lithium isotope ratio determination without mass spectrometry. The Zeeman effect, which involves the splitting of atomic energy levels in the presence of a magnetic field, was utilized to improve spectral resolution between the absorption features of ^{6}Li and ^{7}Li (670.8 nm and 323.3 nm). Experiments used in-house enriched standards (^{6}Li: 7.5–54%) under magnetic fields of 0.2–1 Tesla. At 670.8 nm and 10 ppb Li, no variation in absorbance with isotope composition was observed. However, at 323.3 nm and 3.6 ppm Li, absorbance decreased with increasing ^{6}Li, indicating possible isotope discrimination. A calibration curve of absorbance versus ^{6}Li composition was successfully plotted. This suggests potential for cost-effective isotope analysis. The method is promising where mass spectrometry is unavailable.

3.15. Determination of ultra-trace impurities in semiconductor grade solvents received from Semiconductor laboratory, Punjab

High purity solvents are critical in semiconductor’s manufacturing, where even trace levels of metallic impurities can compromise device performance, lower yields, and cause defects in microelectronic components. In this study, high purity solvent samples-namely isopropyl alcohol (IPA) and acetone were obtained from a semiconductor laboratory for the determination of ultra-trace metallic impurities. Elements such as Al, Ag, Au, Bi, Cd, Cr, Co, Cu, Fe, Ga, K, Mg, Mn, Na, Ni, Pb, Sr, Zn were analyzed using GFAAS in each sample. Extensive temperature program optimization was carried out to achieve enhanced sensitivity and selectivity, enabling detection limits below 10 ppb in IPA and acetone. This work is crucial for maintaining the stringent purity standards required in the semiconductor industry and supports the production of reliable, high-performance electronic devices.

3.16. Determination of iron in various processed biological samples using GFAAS for CDFD, Hyderabad

A total of 393 processed biological samples were received from the Centre for DNA Fingerprinting and Diagnostics, Hyderabad, for their studies on the tracking of genes. Iron analysis in bacterial samples was conducted using GFAAS. Bacterial samples were digested and diluted appropriately before analysis. Following successful calibration, the same optimized furnace program was applied to the bacterial samples. For quality assurance and to monitor matrix effects, standard addition recovery tests were performed for every set of five samples. This approach ensured accurate quantification and helped assess potential matrix interferences, enhancing confidence in the instrumental recovery rates. The combined use of calibration verification, characteristic mass monitoring, and standard addition techniques ensured reliable and precise measurement of iron content in the bacterial samples. Quantification was done using standard addition calibration method. The concentrations of iron were found to be in the range of 0.07-3 ppm.

3.17. Determination of Cs and Rb in acid digests of spodumene mineral for ILCE conducted by AMDER, Bangalore

With rising lithium demand, spodumene deposits are increasingly evaluated not just for Li_2O but also for valuable by-products like cesium (Cs) and rubidium (Rb). Accurate quantification of Cs and Rb is essential for resource assessment. GFAAS method was developed for their determination in spodumene digests. A 0.5 g sample was repeatedly digested with HNO_3 and HF, followed by filtration, ignition, and acid treatment. GFAAS was chosen for its high sensitivity and low sample requirement. Cs determination was complicated by its low ionization potential (3.9 eV), leading to signal loss. Various chemical modifiers, including 0.1% Pd, Pd + Mg, and 0.2% H_2SO_4, as well as Zr and Ir tube coatings, showed limited effectiveness. In contrast, 1% potassium significantly improved atomization, sensitivity, and calibration. Co-injection of 10 µL sample with 10 µL of 1% K provided linear calibration for Cs (10-40 ppb, $R^2 = 0.997$) and Rb (5-20 ppb, $R^2 = 0.999$). The method was applied to real spodumene samples and validated by spike recovery, yielding 6.2 ± 0.7 ppm Cs and 13.0 ± 1.1 ppm Rb.

3.18. Development of *cw*-laser excited atomic fluorescence technique and its application for the determination of Rb and Na in real samples

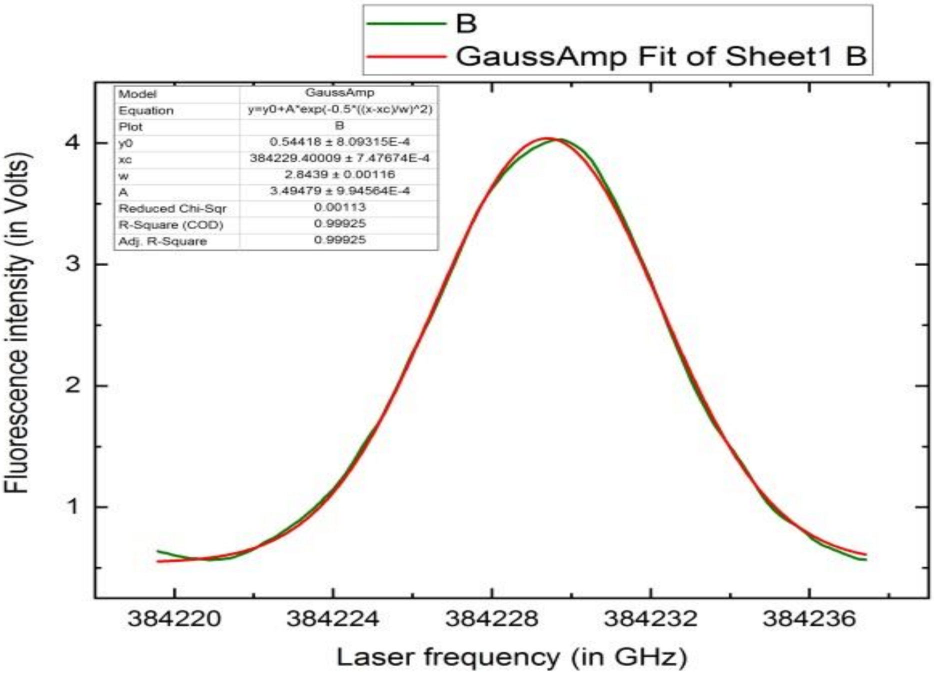
Studies to develop a *cw*-laser-excited atomic fluorescence spectrometry (LEAFS) technique were carried out to exploit the extreme high sensitivity of this method for analysis of real samples. LEAFS is based on the excitation of gaseous atoms by optical radiation tuned to the resonance transition of the atoms of interest and the measurement of the resultant fluorescence radiation. The high selectivity by the laser radiation tuned to the analyte of interest and high sensitivity are the significant advantages of this technique. Further, lower spectral interferences compared to Atomic Emission Spectrometry (AES) is possible in LEAFS. However, the technique can be utilized to analyze one element at time, and hence has a drawback compared to simultaneous multi-element technique. Studies to optimize signal (SNR) to noise ratio dependence on the laser power were carried out. Two different modes of signal detection were investigated to minimize the consumption of sample and time for sample analysis.

3.19. Laser studies to enhance fluorescence intensity of Rb and Na at saturation conditions of atomic transitions

Studies were carried out to optimize the laser power to improve the SNR as it is very well known that laser intensities are sufficient to saturate the atomic transition. As the larger photon emission per individual excited atom is feasible, the trace levels of atomic concentrations can be efficiently determined. Therefore, the power and beam diameter and the wavelength of the excitation radiation were optimized to improve the SNR. Best SNR was achieved at a power of 100 mW and the beam diameter of ~ 5 mm. It was observed that the position of the exciting laser beam in the flame was not extremely critical due to the large beam diameter of ~ 5 mm. Various light traps, apertures, and light baffles were utilized such that the background noise due to scatter was significantly reduced. By operating close to the saturation conditions of the transition; larger signal levels and higher linear dynamic range was achieved.

3.20. Laser studies on the acquisition of Rb fluorescence signal by standard wavelength scan

The general mode of acquisition of the fluorescence signal in *cw*-LEAFS technique is carried with lock-in detection by amplitude modulation of the laser beam while the laser frequency is scanned across the atomic resonance. Furthermore, by scanning the laser frequency (wavelength) across the collision and temperature broadened atomic transition in the flame, the interference from non-specific particles within the flame is corrected. The typical fluorescence spectrum observed as the laser scanned across the resonance is shown in figure 1. The width of the spectrum from the fitting was estimated to be ~ 7 GHz and is consistent with the typical spectral widths of atomic lines observed in flames. The amplified signals from the lock-in-amplifier were recorded by a digital storage oscilloscope as function of laser frequency (wavelength).



Model	GaussAmp
Equation	y=y0+A*exp(-0.5*((x-xc)/w)^2)
Plot	B
y0	0.54418 ± 8.09315E-4
xc	384229.40009 ± 7.47674E-4
w	2.8439 ± 0.00116
A	3.49479 ± 9.94564E-4
Reduced Chi-Sqr	0.00113
R-Square (COD)	0.99925
Adj. R-Square	0.99925

Figure 1: The fluorescence spectrum (6 averages) of ~ 500 ppb concentration of Rb solution from air-acetylene flame recorded by scanning the laser about the resonance of Rb D2 line.

3.21. Studies on the analytical calibration of Rb signals using wavelength scanning mode

The foremost and preliminary step to adopt the LEAFS technique for sample analysis is the calibration plot for series of standard solutions. Therefore, the fluorescence spectra of Rb standards in the range of 100-400 ppb were recorded by scanning the laser frequency across the resonance of Rb D2 line and shown in following figure 2. From figure 2, it can be very clearly observed that for the case of standard blank no fluorescence peak structure is observed. This further establishes that the signal due to true resonance fluorescence emission from Rb atoms aspirated into the flame is recorded. Moreover, it ascertains the signal is not due to any scattered radiation from the species in the flame. However, this method causes significant loss of the sample solution as well as requires relatively longer time for recording the signal. A relatively slow scan of *cw* laser ensures a smooth scan across the resonance profile of the transition without any mode-hops. The variation of the laser power while scanning the laser frequency across the spectrum of the atomic transition causes additional problem of a non-linear background. Furthermore, precision and detection limits become poorer because of the short time the intensity maximum signal is observed during a wavelength scan (i.e., the noise reduction advantage of a relatively longer RC time constant is lost).

Hence, the fluorescence acquisition was carried out by stabilization of laser frequency to an atomic resonance.

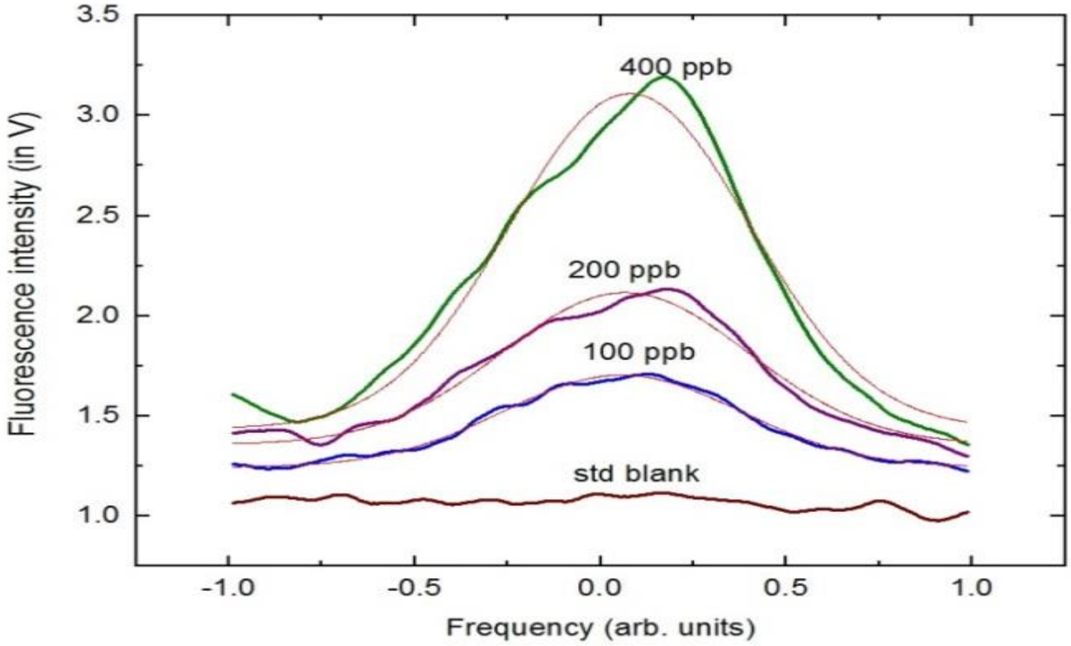


Figure 2: The fluorescence spectrum of Rb standard solutions along with acid blank.

3.22. Stabilization of laser frequency to improve the precision in fluorescence signal acquisitions

The wavelength scan method utilizes the sample nebulizer of a commercial FAAS, it causes significant loss of the sample solution as well as requires relatively large time for recording the signal (~180 s). The typical sample uptake rate or nebulization rate is ca. 8 mL/minute; however, this can vary depending on the specific instrument and nebulizer design, with most systems utilizing a sample uptake range between 2-10 mL/minute. The analytical signal acquisition time is critically dependent on the wavelength scan range, rate and time constant of the lock-in amplifier. Hence, in this mode the measuring time is too long and larger sample volume is needed to attain the true fluorescence signal. One of the major parameters that plays a significant role in trace and ultra-trace level analysis is the sample volume of the analyte required for carrying the analysis. From the perspective analyst, a technique or method which utilizes lesser amount of sample volume is always advantageous. Therefore, detection of fluorescence at fixed laser frequency is required and hence the necessary development is carried out to lock the laser frequency to an atomic resonance.

In this method of detecting the fluorescence at a fixed resonance frequency, a small portion (few 100mW) of the laser output beam of *cw*-Ti:Sa laser tuned to the resonance of Rb D2 line was sampled and was directed into a commercial sealed pyrexRb gas cell. The fluorescence emitted as the laser wavelength is scanned across the resonance of Rb D2 transition is detected in a direction transverse to the laser beam propagation by using a photo-multiplier tube. A small sinusoidal voltage signal of 6 to 10 mV at a frequency (f_m) of 70 to 100 Hz from a commercial lock-in-amplifier was mixed with the external voltage ramp signal that is fed to the Brewster plate using a home-built adder circuit. The magnitude of small sinusoidal voltage is determined by carefully observing the depth of frequency modulation of the laser frequency from the wavemeter reading. By demodulating the fluorescence signal at the modulation frequency (f_m), the first derivative frequency discriminate error signal with zero-crossing at the peak of the fluorescence signal is obtained. In order to lock the laser frequency to the Doppler broadened hyperfine component, the first derivative signal was sent to an electronic servo (PI) and fed back to the Brewster plate in a standard frequency-control loop. After, the laser was locked to the hyperfine component; the corresponding absolute frequency value was measured using wavemeter. The full width at half maximum (FWHM) of the fluorescence peaks due to Doppler broadening corresponding to the room temperature was estimated to be ~250 MHz. A plot of the rms noise of the dispersive error signal when the laser frequency is locked to the major hyperfine component is shown in following figure 3. From the measured rms noise of the error signal, the precision of the measured frequency is found to be ~800 kHz.

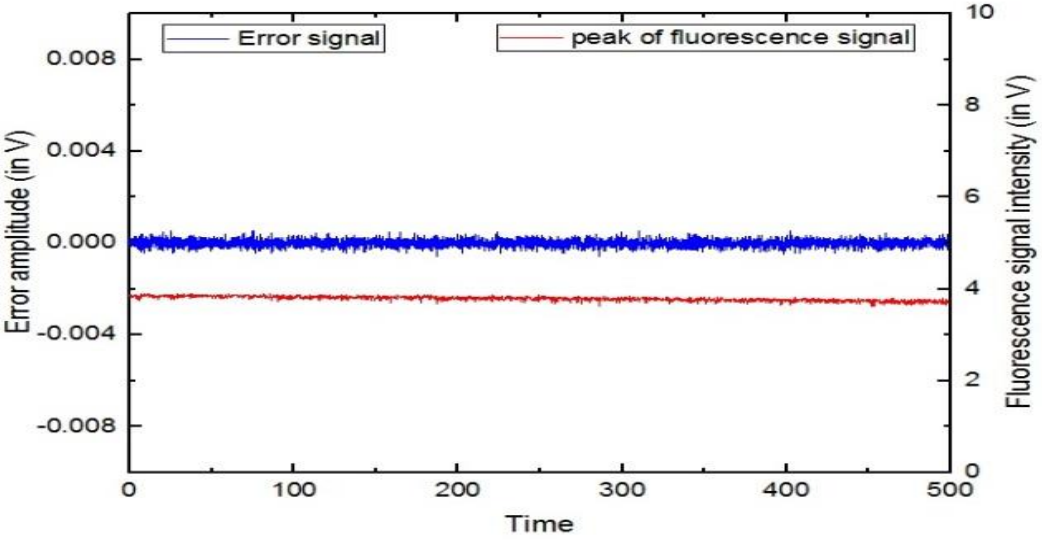


Figure 3: The frequency stability for about 500 s of the laser locked to F=3 to F= (4,3,2) hyperfine component of ^{85}Rb isotope.

3.23. Rb fluorescence signal acquisition after stabilizing the laser frequency to ^{85}Rb hyperfine component

With precise stabilization of the laser frequency to one of the major hyperfine components in Rb gas cell, the laser frequency is now fixed on to the peak of the atomic resonance. About ~10 mW of this laser beam with laser frequency locked to one of the hyperfine components, was modulated at 1000 Hz and directed along the 10 cm length of air-acetylene flame of AAS. The fluorescence intensities were obtained to be a plane DC signal (Figure 4) unlike the Gaussian spectrum that is observed during the frequency scan mode of analysis. By measuring the fluorescence intensities of various standard solutions, it was observed that in this frequency locking technique better precision and calibration was achieved.

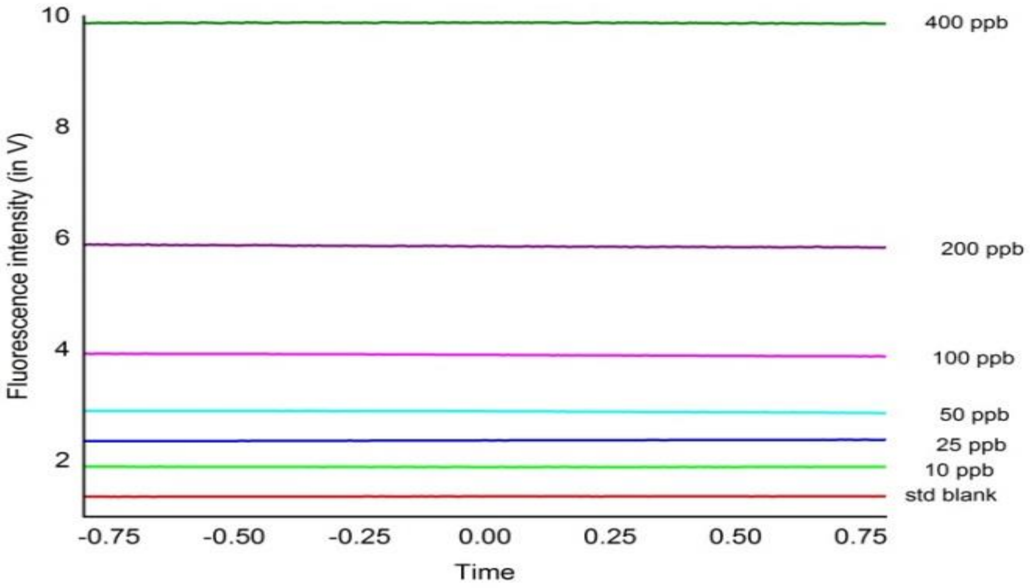


Figure 4: The fluorescence DC values for different standard solutions of Rb observed when the laser was locked to one of the hyperfine components.

3.24. Quantification of Rb in Spodumene SRM by LEAF technique

To establish the accuracy of any technique for its application to real samples, it must reliably reproduce the results of a standard reference material or a certified reference material. Therefore, the spodumene SRM (NBS-181) solutions in duplicate along with its process blank were obtained after digestion as per the standard procedure. 2% HNO_3 solutions of high pure water was used as a standard blank solution for all the experiments. These solutions were further diluted as per the requirement using high purity water. The average value (114 ± 6 ppm) determined by LEAF technique in a fixed frequency mode is in good agreement with the certified value (109 ± 3.6 ppm) in the SRM/NBS-181 of spodumene.

3.25. Studies to obtain *2f*-wavelength modulation spectroscopy for Rb and Cs in gas cells

The high spectral purity of single-mode *cw* lasers dramatically simplifies the spectral isolation of the absorption signal. There is no requirement for a monochromator, which is necessary with hollow cathode lamps (HCL) in order to isolate the characteristic analytical line from other spectral lines of the HCL, e.g., buffer gaslines or lines from other elements sputtered from the cathode. In addition to high spectral purity, by additional wavelength the detection limits in atomic absorption spectrometry have been significantly improved. Hence, by modulating the wavelength of the laser, the absorption for Rb D2 line along with its second harmonic (*2f*) from a dual phase lock-in-amplifier was investigated. The signals from the photo diode detector were processed by a lock-in amplifier in *2f* mode. The signal from the photo diode detector was found to yield a peak absorption value of ~ 6.5 x 10^{-4}V while, the magnitude of the *2f*-signal amplitude was found to be ~5.2 V. The signal was improved by a factor of ~ 8000. Signal to noise ratio of both *1f*-signals and *2f*-signals for Cs were found to be 3000 and 1000 times higher than the direct absorption signal from the photo-detector respectively. Further experiments are being carried out to implement this method in flames or micro-plasmas for sample analysis.

3.26. Two step excitation studies in rubidium to investigate the structure of excited $^2D_{5/2,3/2}$ levels

The structure of the excited states of Rb atoms is investigated by step-wise excitation using two lasers. The technique of crossed atomic beam fluorescence was adopted to lock the first excitation laser to a hyperfine component of Rb D2 transition by modulating the laser with an acousto-optic modulator. A portion of this frequency stabilized laser beam was counter propagated with the beam from another laser which was tuned to the resonance of the second excitation transition in a commercial Rb vapor cell. The (blue) fluorescence from second excited step was recorded to observe the structure of excited $^2D_{5/2,3/2}$ levels.

3.27. Experimental studies to measure the hyperfine frequency separations in Rb $5S_{1/2}$ - $5D_{5/2}$ transition in a vacuum chamber

The rubidium vapor was generated in a quartz crucible heated to temperature of about 825 K. The output beam of the laser from *cw*-ring Ti:Sa laser was sent to the atomic beam apparatus where it was focused to 50 μm diameter by a lens, it was then retroreflected back to generate counter-propagating beam to eliminate 1st order Doppler effect. Frequency scan of the fluorescence spectra was calibrated by passing the laser beam through a broadband electro-optic phase modulator. The separations between the major four hyperfine components were measured and were found to be 4.8(2), 4.4(2) and 3.9(2) MHz.

3.28. Studies on the design of horizontal anode-cathode (HAC) electrode system for collection of ions in Laser optogalvanic setup in flames for LEI

Laser-enhanced ionization (LEI) is a very sensitive trace analysis technique which is based on the spectrally selective laser excitation of analyte atoms followed by their collisional ionization and detection in a suitable atom reservoir. Flames of air-acetylene and hollow cathode discharges are good source for atoms. Experimental studies to configure and optimize this technique) in flames are being carried out for trace elemental analysis. To acquire the signal in LEI system, a horizontal anode-cathode (HAC) electrode system comprising of two SS plates was designed and fabricated. Currently, testing of the electrode system and acquisition of ion signal by lock-in-detection is in progress.

3.29. Quantification of sodium in spodumene samples by Flame Atomic Absorption Spectrometry (FAAS)

The sodium content in Spodumene samples were received for inter-laboratory comparison exercise (ILCE). The samples were analyzed in Flame Atomic Absorption Spectrometry. A good calibration curve for the standards was achieved. About 15 samples were analyzed for the sodium content.

3.30. Synthesis and characterization of gum kondagogu stabilized zinc oxide nanoparticles and its application as an antibacterial agent against wastewater borne bacteria

Wastewater recycling is one of the viable options to attain sustainable management of water. Wastewater is a rich source of heterotrophic bacteria, total coliforms, E. coli, fungi etc. and microbial disinfection is necessary for its reuse. In this context, tree gum capped zinc oxide nanoparticles (ZnO NP) were utilized as an antibacterial agent towards disinfection of wastewater bacteria. ZnO NP were synthesized employing a simple alkaline precipitation with sodium hydroxide using zinc acetate as a metal precursor and gum kondagogu as a stabilizing agent at different concentrations (0.1-0.5%). Synthesized NP at optimal gum condition (0.5%) was characterized using UV-visible absorption spectroscopy (UV-vis), dynamic light scattering (DLS), zeta potential, X-ray diffraction (XRD), Fourier transform infrared spectroscopy (FTIR) and transmission electron microscopy (TEM) techniques.

ZnO NP showed an absorption maximum at 351 nm in the UV-vis; zeta potential value of - 35 mV and z average value of 254 nm in DLS. XRD pattern of ZnO NP showed distinctive diffraction peaks at 31.7°, 34.4°, 36.2°, 47.5°, 56.6°, 62.9°, 66.3, 68° and 69° corresponding to (100), (002), (101), (102), (110), (200), (112) and (201) planes of the characteristic hexagonal wurtzite crystal structure of ZnO. The FTIR data indicated the binding of zinc ions with hydroxyl, protein's amide I, carboxylate and carbonyl functional groups of the gum. The IR data on metal binding, stabilization and capping of the NP by various functional groups of gum is further supported with the analyzed biochemical gum composition. The total reducing sugar and protein content in 0.5% gum solutions were 1732.14 ± 1.6 µg/mL and 42.2 ± 0.4 µg/mL, correspondingly. The size of the NP ranged from 12.4 nm to 35.7 nm and the mean particle size was 24.8 ± 5.1 nm.

The *in vitro* antibacterial action of the synthesized gum stabilized ZnO NP was studied against the wastewater borne bacteria, *Escherichia coli* ATCC 25922 (Gram negative) and *Bacillus subtilis* ATCC 6633 (Gram positive), with resazurin broth assay. The minimum inhibitory concentration (MIC) values of 900 µg/mL and 900 µg/mL and minimum bactericidal concentration (MBC) values of 1800 µg/mL and 900 µg/mL of ZnO NP were recorded towards *E. coli* and *B. subtilis*, respectively. At MIC concentration of 900 µg/mL, the bacterial growth reduction (%) was 98.42 ± 0.5 and 99.99 ± 0 for *E. coli* and *B. subtilis*, correspondingly. The respective MBC values as low as 1800 µg/mL and 900 µg/mL were able to reduce and inhibit bacterial growth by 99.9%. Thus, the current study on ZnO NP mediated bacterial disinfection has wide applications in wastewater treatment and remediation of microbially contaminated effluents.

3.31. Biochemical and molecular identification of waterborne bacteria and fungi isolated from different wastewater sources

A number of waterborne bacteria and fungi (5 No) isolated purified and preserved from different wastewater sources (drainage, STP). NTC1 & NEC2 bacterial strains were tentatively recognized as *Escherichia coli* & total coliform based on colour development on chromogenic media. Isolated genomic DNA was amplified with universal primers for 16S rDNA & sequencing was compared with NCBI GenBank database. Strain NEC2 was confirmed as *E. coli,* based on sequencing and while amplicon of NTC1 failed sequencing.

Based on biochemical characterization, NF1 & NF2 fungal strains were tentatively recognized as *Aspergillus* sp & *Cladosporium* sp. Isolated genomic DNA was amplified with ITS (internal transcribed spacer) 1 & ITS4 primers of fungal 18S rDNA & sequencing of 600 bp PCR product was compared with NCBI GenBank database.

Strains NF1 & NF2 were identified as *Aspergillus niger* & *Cladosporium tenuissimum* based on amplicon sequencing.

3.32. B-10 isotopic abundance determination in boron carbide samples using Particle Induced Gamma ray Emission Spectroscopy (PIGE)

Boron carbide is known for its unique combination of physical and chemical properties, including low density, high hardness, excellent chemical and thermal stability, and high neutron absorption cross-section. The isotopic analysis of boron is important for evaluating its effectiveness as a neutron absorber. However, determining the ratio of boron-10 to boron-11 through chemical methods is often laborious and time-consuming, as the dissolution of boron carbide is very difficult. We have developed and employed a non-destructive Proton Induced γ-Ray Emission Spectroscopy (PIGE) methodology, for the rapid determination of $^{10}B/^{11}B$ isotopic ratio and B-10 isotopic abundance with a high precision.

Several boron carbide (B_4C) powder samples (~ 60 nos.) were received from a private industry, for determination of B-10 abundance. These are the samples of the boron carbide powder meant for export. The powder samples were pelletised into 15 mm diameter. The pellets were mounted on the sample manipulator and placed inside the scattering chamber. The scattering chamber is pumped down to a vacuum of ~ 10^{-6} torr. A well-collimated beam of protons of energy E_0 (3.0 MeV) is impinged on the B_4C pellets. The characteristic γ-rays of 429 keV, 718 keV and 2124 keV energy, from the nuclear reactions $^{10}B(p,\alpha\gamma)^{7}Be$, $^{10}B(p,p'\gamma)^{10}B$ and $^{11}B(p,p'\gamma)^{11}B$ respectively, were detected using HPGe detector. The B-10 isotopic abundance was determined by taking the yield ratios of 718 keV (or 429 keV) and 2124 keV for the sample and CRM standard. The samples were found to have B-10 in natural isotopic abundance. The RSD/precision of the measurements is $\leq$1%. This work has generated a revenue of Rs.15 lakhs. Production of boron carbide CRM for the "Determination of Boron-10 isotopic abundance in boron carbide" is under progress.

3.33. Coincidence Ion Beam Analysis for interference free determination and depth profiling of elements

Coincidence and anti-coincidence measurement techniques are being used in nuclear physics, radioactivity metrology, and in the nuclear analytical measurements. By employing coincidence techniques, it is possible to achieve greater sensitivity, precision, and interference-free determination of elemental composition in nuclear analytical experiments. A coincidence mode Ion Beam Analysis (CIBA) setup is being established. For this, we have employed a high-speed, four-channel digitizer, which eliminates the need for traditional spectroscopy amplifiers, constant fraction timing discriminators, universal coincidence modules, counters, etc. This compact desktop system integrates four independent 16 k-channel digital MCAs for nuclear spectroscopy. Key features of the system include four detector inputs with simultaneous data acquisition, 14-bit flash ADCs with a sampling rate of 100 MS/s per channel, and a time resolution of up to 10 ns. Figure 5 below shows the experimental setup for carrying out CIBA.

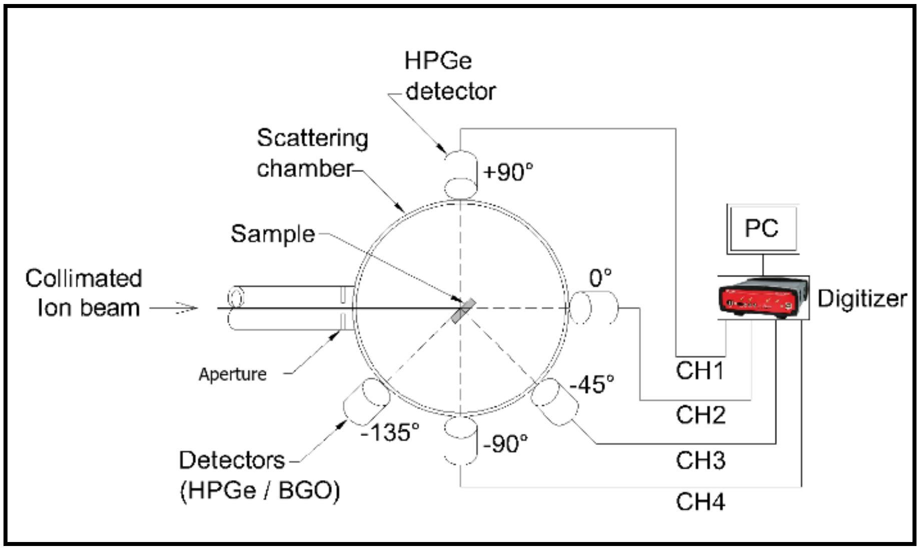


Figure 5: Experimental setup for carrying out CIBA

Using this facility Elastic recoil Detection Analysis of hydrogen in thin polymer foils was carried out in coincidence mode by employing two charged particle detectors placed at 90°apart. The coincidence measurements have eliminated signal from other elements such as C, O.

The facility was also employed for the development of Coincidence-Nuclear Resonance Reaction Analysis (CI-NRRA) methodology for interference free depth profiling of Ni. The resonance at 1424 in ^{58}Ni (p, γ)^{59}Cu nuclear reaction was employed. The characteristic γ-rays of energy 4328, 492 KeV and 4821 keV were detected. The 4328, 492 KeV γ-rays which emit concomitantly were detected in coincidence mode. The angular correlation studies for maximum coincidence events were carried out. The resonances at 1424 keV and 1834 keV were studied in Ni/Si, Cr/Ni/Si and Ni bulk targets. Using CI-NRRA the interference from Cr and Si were eliminated. Figure 6 shows how the CIBA has eliminated the interference from the Cr film while carrying out NRRA.

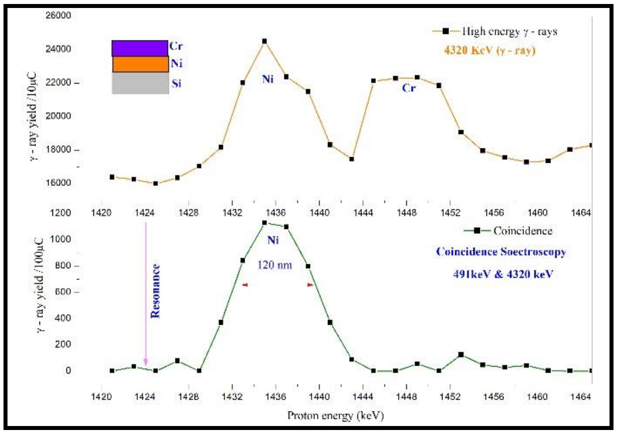


Figure 6: CIBA eliminated the interference from the Cr film while carrying out NRRA.

3.34. Inter diffusion studies of Cr on zirconium-based substrates. Deposition of Cr on zirconium-based substrates by PVD methods and their inter diffusion studies

A work programme was initiated to study the thermal stability of Cr/Zr/Si (or) zircaloy (substrate). In this regard, the samples (Cr(70 nm)/Zr(800 nm)/Si or Cr(70 nm)/zircaloy-4) were deposited by DC magnetron sputtering, were vacuum annealed in 300-600°C temperature range for 1 h. EBS analysis of these samples was carried out using 3.06 MeV α beam. Simulation of the acquired spectra using SIMNRA showed that the as-deposited Cr(film) /Zr(film)/ Si (substrate) samples were containing ~ 10 at.% of oxygen in the entire Cr- layer (70 nm) and 20 at.% of oxygen in the top 40 nm region of the Zr layer of the sample. While, around 2 at.% of Zr diffusion was found into the Si substrate and its thickness was ~ 2 μm. Samples annealed at 600°C became non-adherent and got peeled off from the Si substrate. It was found that out diffusion of Zr into Cr was found in the samples annealed above 500°C and the oxygen content on the top layer (50 nm) was ~ 50 at.%.

In continuation of the studies on inter diffusion of Cr in zirconium-based substrates, another set of Cr/Zr/Si and Cr/zircaloy-4 were prepared by DC magnetron sputtering, and vacuum annealed at 320°C temperature for different (1 h, 6 h, 12 h and 24 h) time durations. RBS analyses of these films showed that diffusion of Zr is observed in the films annealed for more than 6 h at 320°C.

3.35. Determination of H, and D in Zr-2.5Nb samples by Ion Beam Analysis (IBA)

ERDA and p-EBS measurements were performed on the four Zr-2.5Nb samples treated in D_2O and H_2O environments at different temperatures. Analysis of samples by ERDA using 8.0 MeV carbon beam showed that the content of hydrogen and deuterium varied from 2 at.% to 10 at.%. The analysis results of p-EBS experiments carried out using 2 MeV protons, showed that the thickness of the oxide layer varied from 1.5 μm to 2.0 μm. These samples were received from PIED, BARC.

3.36. Thickness determination of coatings on passive catalytic recombiner devices PCRD by RBS

SS wire mesh specimens used in passive catalytic recombiner devices (PCRD) for hydrogen mitigation in nuclear power plants were received from Chemistry Division, BARC, Mumbai and CAD, ECIL, Hyderabad for their surface characterisation. Rutherford backscattering spectrometry (RBS) measurements were carried out, on un-used and used (placed inside reactor building) wire-mesh specimens, by focusing the 4.0 MeV proton beam of 0.3 mm diameter on the wire-mesh strands. The well separated backscattered signals from Pd and Pt coatings on the wire mesh samples were detected. The Thickness of the coatings ranged from 2000 to 5000 Å. The thickness of the Pd and Pt coatings was compared between used and un-used specimens. The RBS spectra of as prepared (A-026) and dry tested in KAPP-4 wire mesh is shown in figure 7.

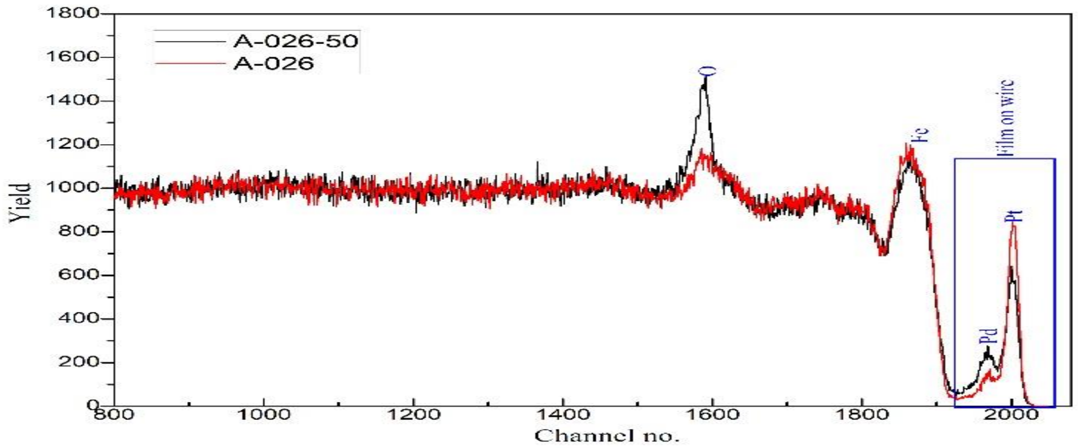


Figure 7: RBS spectra of as prepared (A-026) and dry tested in KAPP-4 wire mesh.

3.37. Depth proofing of H, N and O in cracked and un-cracked regions of Ti-6Al-4V alloys

Ti-6Al-4V alloys are used in space applications. Few numbers of these specimens were received from VSSC/MIDHANI, Hyderabad. Samples were containing several cracked regions. In order to investigate the cause of pitting/cracking on the specimens, the depth profiling of H, N and O were carried out by IBA techniques at cracked and un-cracked regions. H(^{19}F, $\alpha\gamma$)^{16}O, ^{15}N(H, $\alpha\gamma$)^{12}C and ^{18}O(p,α)^{15}N nuclear resonance reactions were utilised for depth profiling of H, N and O respectively. The specimens were probed for more than 1.2μm depth. All the specimens have shown similar nitrogen and oxygen depth profiles in un-cracked and cracked regions, however, only one of the specimens had shown (Figure 8) higher hydrogen concentration profile in cracked region as compared to un-cracked region

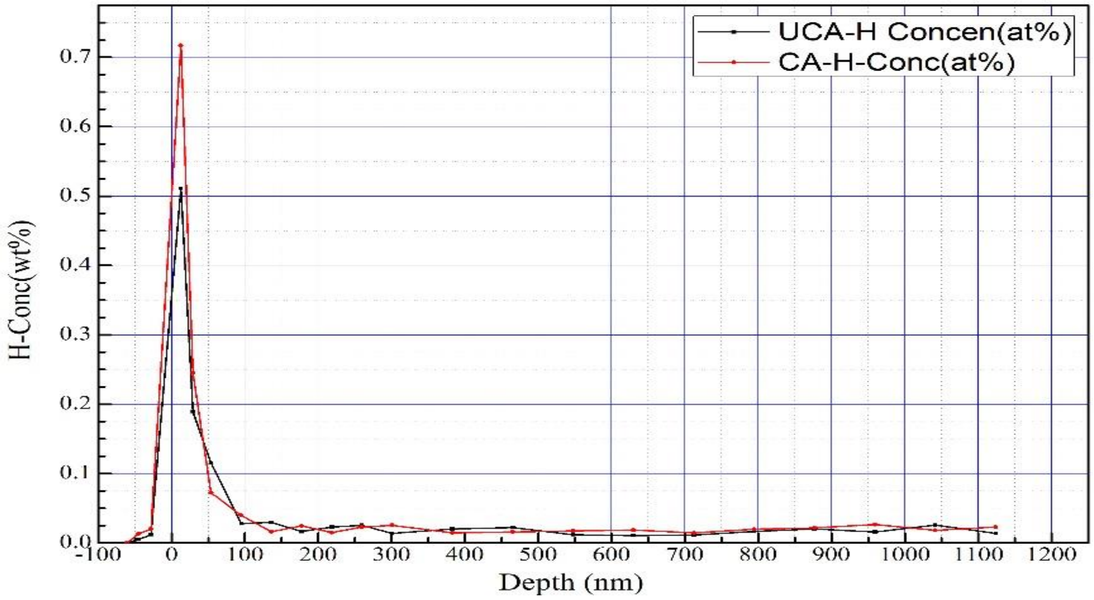


Figure 8: Hydrogen depth profile of one of the specimen in cracked and un-cracked regions of Ti based alloy specimens.

3.38. Determination of thickness and depth profiling of N, O, Zr in ZrN hard coating by IBA

Reactive magnetron sputter deposited ZrN films on Si and SS substrates, (10 nos.), received from MP&CED-BARC were analyzed by RBS and NRRA. The RBS measurements have shown that the films were having a non-uniform distribution of Zr, nitrogen and oxygen across its thickness. The nitrogen depth profiling using NRRA also have shown the non-uniform distribution of nitrogen across the film thickness. Figure 9 shows the concentration profile of one of the ZrN film extracted using RBS and NRRA measurements. The thickness of the films was found to be ~ 1.5 μm.

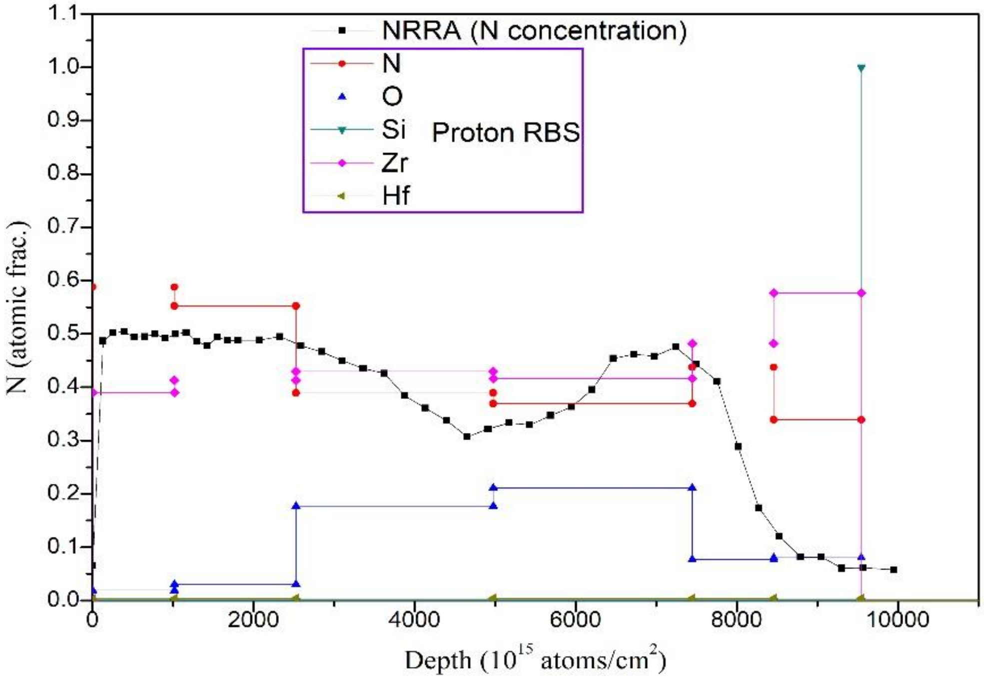


Figure 9: The concentration profile of one of the ZrN film extracted using RBS and NRRA measurements

3.39. Characterisation of VO_2 films for their thickness, phase and morphology

RBS, AFM and GIXRD measurements were carried on sputter deposited VO_2 and Ag-VO_2 films on Si substrates. These samples were received from AMPD, BARC, Visakhapatnam. Thickness and composition of the films were determined using 1.5 MeV α-RBS experiments (Figure 10). It was found that the thickness of the films ranged from 80 nm-165 nm and composition of V and O were in the range of 22-38 and 78-62 at.%. In Ag doped specimens Ag is found to be around 5%. The films were found to be amorphous in nature. From AFM measurements it was found that the RMS roughness was around 0.64 nm.

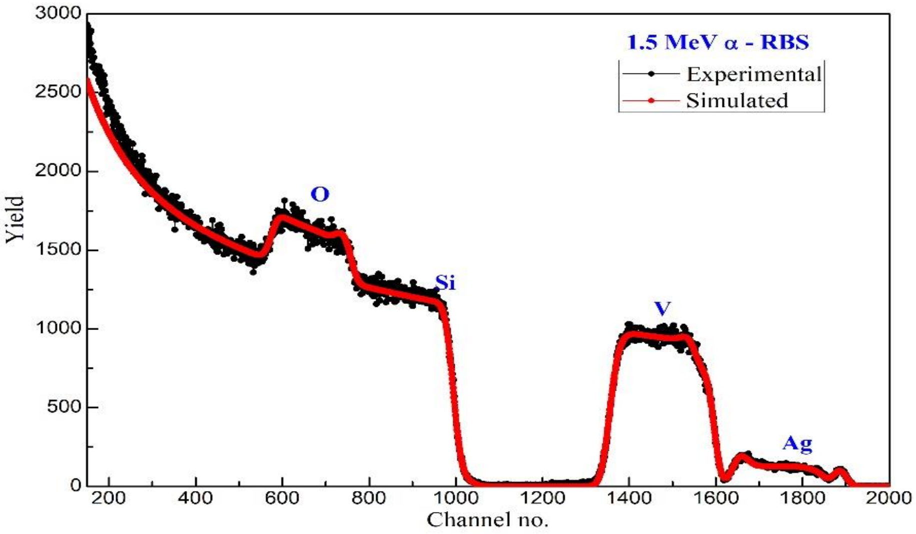


Figure 10: RBS spectra of VO_2 films

3.40. Implantation of Ni in additively manufactured SS 316 and Inconel specimens

Ni^{3+} ions of 4.0 MeV were implanted on additively manufactured SS 316 and Inconel 718 (Wrought, As-built, Solution annealed) specimens (5 nos.). An approximately 300 nA of Ni^{3+} beam current was maintained over a diameter of 6.0 mm on the substrates. A total fluence of 2.0×10^{16} particles/cm^2 were implanted in the specimens. RBS, AFM, SEM/EDS and XRD measurements were carried out on post-irradiated samples.

3.41. Determination of elemental composition in medicinal plant samples by Proton Induced X-ray Emission spectroscopy (PIXE)

Proton Induced X-ray Emission spectroscopy measurements (PIXE) were carried out on medicinal plants (7 nos.), from Punjab university. The powder samples were pelletised and loaded into the scattering chamber. The samples were irradiated with 2.5 MeV protons and the emitted characteristic X-rays were detected with Si(Li) detector. The samples contain S, Cl, K, Ca, Ti, Mn, Fe, Cu and Sr.

3.42. Determination of carbon in steel samples using PIGE

Different steel samples and NIST standards, from RCD, BARC were analyzed for their carbon content using alpha induced gamma emission spectroscopy. The determination of carbon was carried out using $^{12}C(\alpha, \gamma)^{16}O$ and $^{13}C(\alpha, n\gamma)^{16}O$ nuclear reactions which emits 6.1 MeV characteristic γ-rays. These γ-rays were detected using BGO detector.

3.43. Determination of thick target yields of Pt-group and coinage group elements by PIGE

A work programme has been initiated to obtain the thick target yields of coinage and Pt-group elements. In this regard, metallic targets such as Au, Cu and Pd were impinged with 1-4 MeV proton beam and emitted prompt γ-rays were measured using a high purity Ge (HPGe) detector. Analysis of data is in progress.

3.44. Determination of lithium by $^{7}Li(p,\alpha)^{4}He$ nuclear reaction analysis

A methodology is initiated to determine the content of lithium, in several Li containing materials such as spodumene and in a number of materials related to lithium-ion battery, by $^{7}Li(p,\alpha)^{4}He$ nuclear reaction. The reaction is induced by 600 keV protons while the α-ejectiles are detected at 150° emission angle by a surface barrier detector. About 10 μm thick mylar was used as a stopper foil to prevent the entering of backscattered protons and the α-ejectiles into the detector, from other elements such as B, N, O or F through their (p,α) reactions. In this regard, experiments were carried out on lithium carbonate, lithium cobaltate, lithium titanate and few spodumene samples.

4. Quality Control Activity and NABL Accreditation

4.1. ISO 17025:2017 Accreditation – Testing Activities

Chemical metrology is an essential component of progress in industrial development of any nation. The National Centre for Compositional Characterisation of Materials (NCCCM), a division under Chemistry Group, is accredited for ISO 17025:2017 as a Testing Laboratory since 2009. NCCCM management is committed to provide analytical data of highest quality using validated and appropriate methods for analysis and state-of–the-art instruments. It ensures consistent operation to meet customer needs. NCCCM provides specialized analytical services for the determination of constituents down to parts per billion levels in samples of different matrices, meeting the requirements of ISO 17025:2017 standard, which deals with the general requirements for the competence of testing and calibration laboratories. This objective is being catered to different government and private organizations.

Scope of Accreditation (ISO 17025:2017):

S. No.	Category	Matrix	Testing Parameter
1	Water	Water	pH, alkalinity, conductivity, Ca, Mg, Total hardness
2	Residues in water	Water	Cd, Cu, Pb, Mn, Hg, B, Fe, Zn
3	Ores & Minerals	Quartz	Al, Ca, Fe, Mg, K, Na, Ti
		Dolomite	CaO, MgO, Al_2O_3, Fe_2O_3, BaO, SrO
		Bauxite	Al_2O_3, Fe_2O_3, Cr_2O_3, SiO_2, TiO_2, MnO, MgO, V_2O_5, LOI
4	Metals & Alloys	Cast Iron	P, Si, Mn, Ti
5	Residues in Food products	Noodle Powder	Pb
		Tea Powder	K, Mg, Ca, Sr, Ba, Al, P, Mn, Fe, Cu, Zn, As, Cd, Hg, Pb
		Wheat Flour	Fe, Zn

The accreditation is given for a period of two years and is renewed at the end of 2nd year. The on-site surveillance assessment of the testing activities under the scope of ISO 17025:2017 was conducted in June 2024 by NABL appointed technical assessors, for the extension of accreditation. The non-compliances raised during the assessment were closed by submitting the required documentary evidences to NABL. Subsequently

NABL has extended the accreditation status for a further period of one year, until June 2025.

4.2. ISO 17034:2016 Accreditation – Reference Material Producer

In the year 2018 NCCCM got the accreditation for ISO 17034:2016 as a Reference Material Producer (RMP) by NABL, New Delhi. The NCCCM management has committed to ensure and maintain quality aspects of Certified Reference Material production, storage and distribution and has documented policies and objectives. Its objective is to produce Certified Reference Materials (CRMs) for its intended use and conforming to ISO Guide 35 (statistics) and 31 (contents of certificates, labels) which will satisfy the customers and meet their needs. Materials produced will be of international quality and its process of production will meet the requirements of ISO 17034:2016 standard. NCCCM is the only laboratory in the Department of Atomic Energy to be accredited for ISO 17034:2016 by NABL, as a Reference Material Producer.

After successful completion of the on-site assessment in December 2023, the ISO 17034 accreditation status was renewed for a further period of two years from 03/02/2024 to 02/02/2026. Three new CRMs viz., Dolomite, Bauxite and Tea Powder were included in the existing scope of accreditation during the renewal.

The desktop surveillance of ISO 17034:2016 accreditation, for continued compliance in the preparation of reference materials is due for renewal in February 2025. The application process for extension of accreditation was initiated in December 2024 on the NABL portal. Valid documentary evidences for the scope of RMP activities, for continued compliance as per ISO 17034 standard was submitted to NABL.

Scope of Accreditation (ISO 17034:2016):

S. No.	**CRM**	**Testing Parameter**
1	Quartz	Al, Ca, Fe, K, Mg, Na, Ti
2	Noodle Powder	Pb
3	Dolomite	Al_2O_3, BaO, CaO, Fe_2O_3, MgO, SrO
4	Bauxite	Al_2O_3, Cr_2O_3, Fe_2O_3, MgO, MnO, SiO_2, TiO_2, V_2O_5, LOI
5	Tea Powder	Al, Ba, Ca, Cd, Cu, Fe, Hg, K, Mg, Mn, P, Pb, Sr, Zn

4.3. Internal Quality Control Activity

4.3.1. Management Review Meetings and Internal Audits

Management Review Meetings and Internal Audits are conducted periodically as per the requirements of the ISO 17025 and ISO 17034 standards. The outcomes of the internal audits, policies, procedures, change in the scope of work, resource requirements, non-compliances and other improvements were discussed in the management review and the minutes of the meeting was prepared. The experimental and instrumental record books are checked periodically and updated.

4.3.2. Participation in Proficiency Testing (PT)

The laboratory regularly participates in proficiency testing programs and inter-laboratory comparison exercises, as per the requirements of ISO 17025 and ISO 17034 standards, demonstrating the competence in the scope of testing and reference material production. Our Centre participated in the Proficiency Testing Programs conducted by Global PT Provider Pvt. Ltd., New Delhi and Farelabs Pvt. Ltd., Gurgaon on Cast Iron and Tea Powder respectively. The z-score values of all the testing parameters were satisfactory (|z| < 2), except for Hg in Tea powder.

Proficiency Test Results of cast iron and tea powder

Proficiency Test sample	**Testing Parameter**	**z-score**
Cast Iron	Mn	0.70
	P	-0.16
	Si	-1.48
Tea Powder	Pb	0.32
	Cd	1.94
	Hg	3.59
	Fe	0.00
	Zn	-1.91
	K	-0.04

The corrective action was taken by repeating the analysis for Hg and action taken report prepared as per NABL requirements. The result was in close agreement with the value reported earlier to the PT provider. The final PT report of the tea powder sent by the PT provider, showed almost 30% variations in the Hg values reported by the five participating laboratories. The result of the repeat analysis has been communicated to the PT provider by email, seeking their reply.

4.3.3. Participation in Inter-laboratory Comparison Exercise (ILCE)

i) Determination of Yb, La, Gd, Ho, Tm, Er, Tb, Dy, Sm, Eu, Ce, Pr, Nd and P in Monazite. (ILCE conducted by AMDER, Hyderabad).

ii) Determination of Fe, Mn, Ti, Ba and Al in Sillimanite (ILCE conducted by AMDER, Hyderabad).

iii) Analysis of spodumene sample for Li, Al, Fe and Na received from AMDER, Bengaluru.

iv) Determination of Na_2O, CaO, Fe_2O_3, TiO_2, MnO, MgO, Ga_2O_3, V_2O_5 and LOI in candidate alumina CRM (ILCE conducted by NCCCM-BARC).

z-score of NCCCM-BARC for Alumina CRM

Analyte	**Reported values** (% m/m)	**Certified values (% m/m)**	**Robust SD**	**z-score**
Na_2O	0.261	0.271	0.0128	-0.78
CaO	0.0333	0.031	0.0059	0.39
Fe_2O_3	0.00951	0.0099	0.00058	-0.67
TiO_2	0.00443	0.0047	0.00057	-0.47
Ga_2O_3	0.00594	0.0057	0.00037	0.65
V_2O_5	0.0014	0.0014	0.00009	0.00

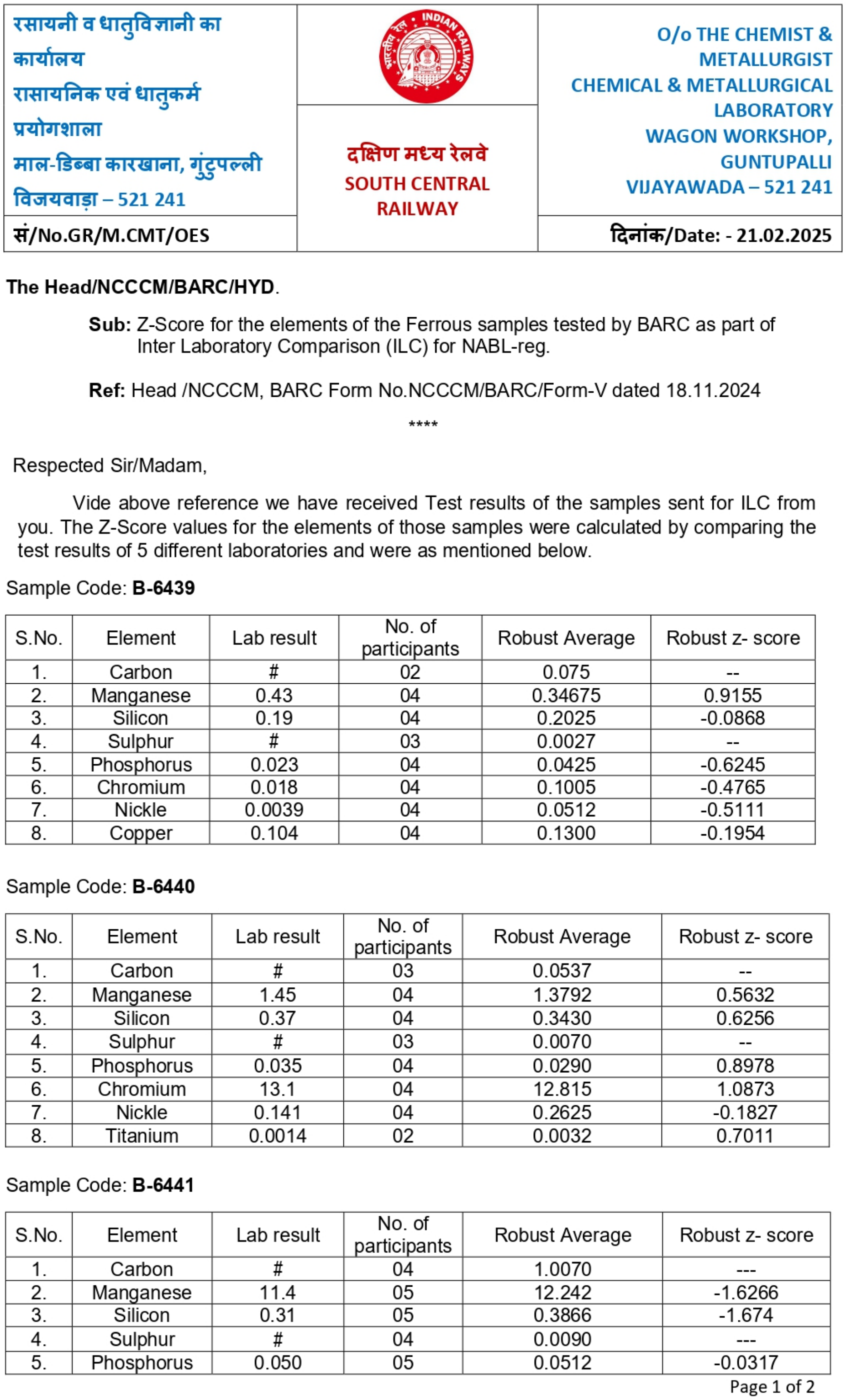
v) Determination of trace impurities in candidate HSLA steel CRM (ILCE conducted by NCCCM-BARC).

z-score of NCCCM-BARC for HSLA steel CRM

Analytes	Reported values (% m/m)	Certified values (% m/m)	Robust SD	z-score
Si	0.227	0.242	0.0156	-0.96
Ti	0.0130	0.013	0.0010	0.00
V	0.0253	0.025	0.0014	0.22
Cr	0.0222	0.018	0.0024	1.71
Mn	1.209	1.23	0.038	-0.55
Ni	0.78	0.78	0.052	0.00
Cu	0.0084	0.0081	0.0005	0.63
Nb	0.0487	0.050	0.0074	-0.18
C	0.111	0.111	0.0034	0.00
P (UU-Vis)	0.0137	0.0144	0.0011	-0.63
S	0.0051	0.0059	0.0010	-0.80
P (ICP-OES)	0.0148	0.0144	0.0011	0.36

vi) Determination of Mn, P, Cr, Ni and Cu in 6 different steel alloys (ILCE conducted by Indian Railways).

z-score of NCCCM-BARC for railway steel alloys



रसायनी व धातुविज्ञानी का कार्यालय रासायनिक एवं धातुकर्म प्रयोगशाला माल-डिब्बा कारखाना, गुंटुपल्ली विजयवाड़ा – 521 241	दक्षिण मध्य रेलवे SOUTH CENTRAL RAILWAY	O/o THE CHEMIST & METALLURGIST CHEMICAL & METALLURGICAL LABORATORY WAGON WORKSHOP, GUNTUPALLI VIJAYAWADA – 521 241
सं/No.GR/M.CMT/OES		दिनांक/Date: - 21.02.2025

The Head/NCCCM/BARC/HYD.

Sub: Z-Score for the elements of the Ferrous samples tested by BARC as part of Inter Laboratory Comparison (ILC) for NABL-reg.

Ref: Head /NCCCM, BARC Form No.NCCCM/BARC/Form-V dated 18.11.2024

****

Respected Sir/Madam,

Vide above reference we have received Test results of the samples sent for ILC from you. The Z-Score values for the elements of those samples were calculated by comparing the test results of 5 different laboratories and were as mentioned below.

Sample Code: **B-6439**

S.No.	Element	Lab result	No. of participants	Robust Average	Robust z- score
1.	Carbon	#	02	0.075	--
2.	Manganese	0.43	04	0.34675	0.9155
3.	Silicon	0.19	04	0.2025	-0.0868
4.	Sulphur	#	03	0.0027	--
5.	Phosphorus	0.023	04	0.0425	-0.6245
6.	Chromium	0.018	04	0.1005	-0.4765
7.	Nickle	0.0039	04	0.0512	-0.5111
8.	Copper	0.104	04	0.1300	-0.1954

Sample Code: **B-6440**

S.No.	Element	Lab result	No. of participants	Robust Average	Robust z- score
1.	Carbon	#	03	0.0537	--
2.	Manganese	1.45	04	1.3792	0.5632
3.	Silicon	0.37	04	0.3430	0.6256
4.	Sulphur	#	03	0.0070	--
5.	Phosphorus	0.035	04	0.0290	0.8978
6.	Chromium	13.1	04	12.815	1.0873
7.	Nickle	0.141	04	0.2625	-0.1827
8.	Titanium	0.0014	02	0.0032	0.7011

Sample Code: **B-6441**

S.No.	Element	Lab result	No. of participants	Robust Average	Robust z- score
1.	Carbon	#	04	1.0070	---
2.	Manganese	11.4	05	12.242	-1.6266
3.	Silicon	0.31	05	0.3866	-1.674
4.	Sulphur	#	04	0.0090	---
5.	Phosphorus	0.050	05	0.0512	-0.0317

Page 1 of 2

रसायनी व धातुविज्ञानी का कार्यालय रासायनिक एवं धातुकर्म प्रयोगशाला माल-डिब्बा कारखाना, गुंटुपल्ली विजयवाड़ा – 521 241	दक्षिण मध्य रेलवे SOUTH CENTRAL RAILWAY	O/o THE CHEMIST & METALLURGIST CHEMICAL & METALLURGICAL LABORATORY WAGON WORKSHOP, GUNTUPALLI VIJAYAWADA – 521 241
सं/No.GR/M.CMT/OES		**दिनांक/Date: - 21.02.2025**

Sample Code: **B-6442**

S.No.	Element	Lab result	No. of participants	Robust Average	Robust z- score
1.	Carbon	#	04	0.5683	---
2.	Manganese	0.74	05	0.8048	-0.9611
3.	Silicon	0.21	05	0.1972	0.3843
4.	Sulphur	#	04	0.0180	---
5.	Phosphorus	0.020	05	0.056	-0.4471

Sample Code: **B-6443**

S.No.	Element	Lab result	No. of participants	Robust Average	Robust z- score
1.	Carbon	#	04	0.1548	---
2.	Manganese	0.73	05	0.7772	-0.9577
3.	Silicon	0.20	05	0.1836	1.0482
4.	Sulphur	#	04	0.0095	----
5.	Phosphorus	0.019	05	0.022	-1.0607
6.	Copper	0.280	05	0.2376	0.3641

Sample Code: **B-6444**

S.No.	Element	Lab result	No. of participants	Robust Average	Robust z- score
1.	Carbon	#	04	0.9828	---
2.	Manganese	0.95	05	1.0588	-0.6326
3.	Silicon	0.56	05	0.6204	-0.5153
4.	Sulphur	#	04	0.0065	---
5.	Phosphorus	0.025	05	0.0162	1.7192
6.	Chromium	1.50	05	1.4778	0.7828
7.	Copper	0.080	05	0.1050	-0.3813

Note: # indicates that the Laboratory has not reported this parameter, hence performance evaluation not done.

SHAIK KHAJA
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Date: 2025.02.21 16:31:32 +05'30'

CMT/WWS/RYPS

4.3.4. Intermediate checks

Intermediate checks of all the standard operating procedures and equipments in the scope of ISO 17025 accreditation are carried our periodically as per planned schedule to check for their compliance.

4.3.5. Training

Training of personnel on ISO 17025:2017 and ISO 17034:2016 are conducted periodically.

4.5. ISO 17025:2017 assessment activities

Underwent on-site assessment of following SOPs under 17025:2017 conducted by NABL.

1. Standard Operating Procedure for determination of total lead in microwave digested noodle powder using Graphite Furnace Atomic Absorption Spectrometry (GFAAS).
2. Standard Operating Procedure (SOP) and Method Validation for the determination of Ca, K, Mg, P, Al, Ba, Cu, Fe, Mn, Sr Zn by ICPAES; Pb, Cd by GFAAS and As, Hg by AFS in Tea powder.
3. Standard Operating Procedure for determination of oxides of Al, Fe, Si, Ti, Cr, Mn, Mg, V and Zr in Bauxite by ICP-OES.
4. Standard Operating Procedure for determination of seven trace elements (Al, Ca, Fe, K, Mg, Na and Ti) in high purity quartz
5. Standard Operating Procedure for determination of Si, Ti, Mn and P in cast iron using ICP-OES.
6. Determination of major and minor components in Dolomite (ASTM Procedure, C1301-95).
7. Standard Operating Procedure for determination of alkalinity of water.
8. Standard Operating Procedure for determination of hardness of water.
9. Standard Operating Procedure for determination of Pb in noodles by GFAAS.
10. Standard Operating Procedure for determination of Pb and Cd in tea power by GFAAS.
11. Standard Operating Procedure for determination of pH and conductivity in water.
12. Standard Operating Procedure for determination of Mn, Cu, Cd and Pb in water by GFAAS.

5. Analytical services

1. Analysis of geological sample for 15 REEs for Gimpex Pvt Ltd, Chennai.
2. Determination of rhenium (Re) in copper tailings for Materials Processing & Corrosion Engineering Division, BARC, Mumbai.
3. Determination of Na, K, Rb and Cs in lepidolite for Mineral Processing Division - BARC, Hyderabad.
4. Determination of Pb in Cu and Zn in Pb, Pb in Ni, Cu in Bi electrodes for Chemistry Division, BARC, Mumbai.
5. Determination of Na, Fe, Mg, Ca, Si and Ti in high pure alumina for NRTC-NALCO.
6. Determination of Ni, Co and trace impurities (Zn, Fe, Al, Cd, Mg and Mn) in nickel hydroxide, for HBL power systems, Hyderabad.
7. Determination of Ga and Ge in zinc samples collected from Hindustan Zinc Ltd, Udaipur for Mineral Processing Division, BARC, Hyderabad
8. Determination of total and leachable boron, total and leachable calcium, trace elements (Ca, Mg and Si), fluoride and SO_2 concentration in BF_3CaF_2 and $BF_3O(C_2H_5)_2$ samples for Heavy Water Plant, Talcher and ECIL, Hyderabad.
9. Determination of Co and Ni in scrap of alloy obtained from MIDHANI, Hyderabada for HWP, Manuguru.
10. Determination of B and Si in boron sample for Materials Processing & Corrosion Engineering Division, BARC, Mumbai.
11. Purification of quartz powder using chemical leaching for Gimpex Pvt Ltd. Chennai.
12. Determination of Na, K, Ca, Mg, Al, Mn, Li, Fe, Cu, Ti and Zr in raw and purified quartz samples for Gimpex Pvt Ltd, Chennai.
13. Determination of Li, Fe, Cr, Mn, Ni and Cu in Pb-16Li alloy for Institute of Plasma Research, Ahmedabad.
14. Determination of Zn and Fe in water samples for KL University, Guntur.
15. Analysis of 4 waters for z average & zeta potential.
16. Assessed viability & purity of different bacterial & fungal strains
17. Mass multiplied the liquid microbial consortiums of waste decomposer and Kavach (biopolymer producer) using jaggery as a cheap propagating medium towards composting of dried leaf litter of NCCCM garden.
18. Quantification of Li extracted from 18 coal fly ash by GFAAS.
19. Quantification of K in high purity quartz samples by GFAAS
20. Cerium oxide samples (5 nos.) from AMD were analyzed by SEM/EDS for their morphology and qualitative analysis

21. Spin coated CN films (7 nos.), from AMD, were analyzed by SEM/EDS for their morphology and qualitative analysis.
22. CaF_2:BF_3 complex samples (2 nos.) from ECIL, Hyderabad were analyzed by SEM/EDS for qualitative and semi quantitative analysis.
23. Lithium ferrite samples (2 nos.) from GITAM University, Visakhapatnam were analyzed by XRD.
24. Phase analysis of $Ni(OH)_2$ powder samples from HBL Power Systems, Hyderabad was carried (2 nos.) by XRD.
25. Phase analysis of alumina powder samples were carried (7 nos.) by XRD.
26. AFM measurements on zirconium carbide, W-Ta-Nb hard/wear resistant coatings and Cr/Zr bi-layer films were carried out.

6. Instrumentation support activities

6.1 Solving the multiple problems of GD-QMS instrument

GD-QMS had developed detector and other problems. All its sub-systems were tested. +15V DC power supply for preamplifier board was restored. Its regulator IC 7815 was replaced. Then the board had started working. Error controller IC-OP07of 1KV DC power supply was replaced with new one. Then the power supply started working. In preamplifier IC AD 549J (input bias current: 250 fA) was replaced with IC AD549L (60fA). Then the detector board was functioning. Potentiometers were adjusted. Variable resister pot of mass display LED IC and mass slope trim pot were adjusted. Gate valve problem was also solved. With all these, GD-QMS instrument was brought back to working condition.

6.2 Rectification of signal display problem of ratemeter of GD-QMS instrument

The instrument ion signal was observed in scanning mode but not in manual mode (in the ratemeter). After tracing, the signal wires of ratemeter were found to be cut. The wires were reconnected and the signal display problem was solved.

6.3 Repairing of communication problem of Flame Atomic Absorption Spectrometry (GBC, Savant make)

On examination of the instrument having 8 lamp turret, it was observed that there was a communication problem between PC software package and the instrument. After proper communication portal setting the communication between AAS and PC started working. In addition, it was also found that there was a low signal and lot of signal noise. These problems were also rectified by doing proper overlapping between HCL (Hollow cathode lamp) and D2 (Deuterium lamp) beam and even by adjusting slit width.

6.4 Rectification of high voltage cable and gas box leakage problems of GFAAS

There were two problems first one is gas box leakage and second one is High voltage 16 core cable problem. Gas box is computer controlled to allow the required gas output to the furnace and the high voltage cable is a 16-core cable and it controls to 7 hollow cathode lamps. The cable insulation was found to be totally ruptured. The gas box leakages were repaired by using teflon tape and tightening the respective screws. The 16-core cable was replaced with new one carefully and tested the required voltages. Then the instrument is functioning satisfactorily.

6.5 Rectification of booting problem of GFAAS instrument

The 120 GB PATA hard disk of GFAAS was not working with instrument software of 32-bit WIN-XP. As the drivers were not available, the entire data in this old hard disk was copied into a new hard disk using cloning software. With this, the GFAAS could be made operational.

6.6 Rectification of gas leakage problem in microwave synthesis reactor

The Microwave synthesis reactor (Anton Paar Monowave 300) system is a series of high-performance mono-mode microwave reactors designed for small to medium scale microwave synthesis. There was a gas leakage problem in the inlet. The gas inlet connector was replaced and the problem was rectified.

6.7 Rectification of high temperature controller problem of muffle furnace

The temperature controller of Ants Prosys high performance muffle furnace was not working at high temperature,1200°C. The temperature inside the muffle is controlled using a temperature controller, which monitors the temperature and also adjusts the power supplied to the heating elements accordingly. There is a solid-state relay (SSR) between controller and heating element. It was checked and found to be not working properly. After replacing with the new SSR, the furnace was started working at high temperature also.

6.8 Rectification of Chiller temperature problem of Inductively Coupled Plasma-Optical Emission Spectrometry (ICP-OES)

The ICP-OES system (JY 2000) was not working. Then the temperature controller of the chiller was checked and found to be faulty. After replacing with the new temperature controller the chiller temperature got controlled and working satisfactorily.

6.9 Rectification of voltage fluctuation problem of High voltage Power supply (1KV DC) of laser system

The output of the power supply in the laser experimental system was fluctuating that is from 100V to 500V and after checking we could find that 20K variable helipot was the problem. After replacing it the power supply started working satisfactorily.

6.10 Rectification of two problems of orbital shaking incubator

The incubator is designed for precise temperature control and simultaneous shaking applications required in fermentations studies. This instrument has developed two problems. Those are 1) a 4-inch LCD display was not working and 2) Temperature was not getting controlled. The 4-inch LCD display problem was rectified by dry soldering and the Temperature controller problem was rectified by replacing the two SSR (Solid state relay) relays.

6.11 Rectification of calibration problem of Eutech's Cyber Scan pH/Ion 510 microprocessor-based Meter with ATC probe

This instrument was not getting calibrated. It has been checked and found that the probe connected to the instrument was faulty. So, a spare probe was connected and tested the calibration and it was found to be working satisfactorily. Now the instrument is working perfectly.

6.12 Rectification of temperature problem of 4.5 L Bench top freeze dry system

This system was not reaching the set temperature (-40 ºC). When the two compressors were checked, it was found that one of the compressor gases (R-508) was leaked completely. The gas is to be refilled.

6.13 Repairing of SRS lock-in-amplifier

Two of the ICs viz., 7815 and 7915 were found to be short in the lock-in-amplifier and hence there was no output from the preamplifier. After replacing both regulator ICs 7815, 7915 and also preamplifier IC AD645, the lock-in-amplifier could be made functional without any errors.

6.14 Status Display problem of gamma area monitor

The STATUS display of the gamma area monitor was not working. The power supply regulator IC 7805 was found to be faulty. After replacing the IC, the display was restored.

7. General service activities

An amount of Rs.4,33,01,334/- was spent towards vision project, procurement, work orders and AMCs during this review period.

7.1. Vision Project Indents Rs.3,05,08,447/-

1. ICP-OES Ultima Expert - Rs.1,25,00,000/- (Installed)
2. Ion Chromatograph – Rs. 85,00,000/- (Installed)
3. Microwave Digestion Systems (2 nos.) – Rs. 54,00,000/- (Installed)
4. Supply, installation, testing and commissioning of 115 TR capacity chiller package unit for Central Air-conditioning plant – Rs.33,87,391/- (Commissioned)
5. Replacement of valves, strainers, NRVs in condensed and chilled Water lines and associated works in AC Plant – Rs.7,21,056/- (Completed)

7.2. Procurements Rs.70,17,083/-

a) ICP RF Generator (ICP generator and connection kit)-Rs.15,70,698/-
b) Spares for LCMS – Rs.9,11,040/-
c) Surge protection devices (SPDs) – Rs.9,87,660/-
d) HEATEX LCD SMD Rework Station 600 W power consumption - Rs. 2,25,000/-
e) FLUKE hand held 1000 kHz Digital multimeters – Rs.2,29,996/-
f) Purchase of seamless high compressed pressure Argon gas cylinders (40 nos.) to minimize the dependency on other units – Rs. 4,70,466/-
g) Re-filling of Argon gas in empty cylinders (110 nos.) – Rs. 302729/-
h) Procurement of LED tube lights for the replacement the old fluorescent lamps for energy efficiency (330 nos.) – Rs. 1,11,600/-.
i) Procurement of red soil and cow dung for new garden and maintenance of existing garden – Rs. 4,73,652/-.
j) Supply and installation of prefabricated structural arch roof along with supported columns – Rs. 9,89,076/-.
k) Supply-installation-commissioning of CCTV system at NCCCM –Rs. 1,83,490/-
l) 12V SMF Batteries (18AH/42AH/65AH/100AH) – Rs. 4,80,805/-.
m) Miscellaneous items – 80,871/-.

7.3. Work orders Rs.11,74,887/-

a) Water drilling services; Two new borewells were commissioned along with revival of two old jet pumps – Rs.4,63,153/- (Rs.2,13,465 + Rs.2,49,688).

b) Testing of seamless argon cylinders, replacement safety valves and providing valve guards in existing argon gas cylinders – Rs.1,09,000/-.
c) Repair, maintenance and installation in AC plant – Rs.2,09,000/.
d) Replacement of roof sheets in old parking shed and painting work – Rs.82,000/.
e) Machining of APURVA steel for chips and disks – Rs.70,990/-.
f) Miscellaneous work orders – Rs.2,40,744.

7.4. Annual maintenance contracts (AMC) Rs.46,00,917/-

a) Comprehensive annual maintenance contract (CAMC) for 115 TR reciprocating compressor package unit central air conditioning system and associated units – Rs.3,84,503/-
b) CAMC for 115 TR screw chiller package unit for AC plant – Rs.5,54,600/-
c) AMC of general electrical systems /installations – Rs.5,18,256/-
d) AMC for electrical sub- station for complete system – Rs.14,54,000/-
e) CAMC for water purifiers – Rs.61,596/-
g) Annual maintenance contract for AF420 AFS – Rs.1,41,600/-
h) Annual maintenance contract for HJY ICP Ultima-2, ICP-OES – Rs.1,40,585/-
i) Annual maintenance contract for GBC make Savanta model Flame AAS – Rs.88,500/-
j) Annual maintenance contract of JY 2000, ICP-OES– Rs. 1,17,816/-
k) AMC of Eltra carbon sulphur analyzer, Model CS 2000 – Rs.1,18,000/-
l) CAMC of desktop personal computers and printers both single function mono and multifunction mono – Rs. 1,20,000/-
m) AMC of LCMS and Nitrogen Generator – Rs.6,05,871/-
n) AMC of GFAAS, Zeenit 650P – Rs. 2,46,030/-
o) AMC for CEM MARS5 closed microwave digestion system – Rs.49,560/-

7.5. Support activities

- Low energy Magnet power supply 40V/100A of Tandem accelerator issue has been resolved and restored to normal operation.

- Generating voltmeter (GVM) of Accelerator problem has been solved by substituting equivalent power module for proper display of terminal Voltage.

- 51/2-digit digital panel meter has been procured and tested for measuring terminal voltage of accelerator with required resolution.

- Testing of 4-channel Multichannel analyser with newly made signal cables for conducting Co-incidence experiments.

- Isolation transformer of ion source issue has been resolved and restored back in the system
- Peristaltic pump is made functional by replacing the faulty microcontroller unit with an import substitute using 89C52 microcontroller.
- Test tube rotator (Tarson, Roto-Spin) is made functional by rectifying the faults in drive circuit.
- Maintenance of LAN, ANUNET and video conference facility.

8. Publications and other activities

8.1. Technology transfer and training for licensees of technology holders

1. Technology of Fluoride Detection Kit (FDK) for ground water (WT07NCCCM) is transferred to M/s Royal Enterprises, Chennai on 11/03/2024 and M/s Techno Surge Industries, Amravati, Maharashtra on 22/11/2024.

2. Training and evaluation of FDK kit prepared by licensees of technology holders.
   1. Orlab Instruments Pvt. Ltd, Hyderabad.
   2. Plast Surge Industries, Amravati, Maharashtra.
   3. Techno Surge Industries, Amravati, Maharashtra.

3. Supply of FDK reagents to NFC, Hyderabad.

8.2. Journal Publications

1. Determination of rare earth elements in ferrocarbonatite using ICP-OES and ICP-MS
   S. Thangavel, G. Venkateswarlu, A. Durga Prasad, B. Sunil Kumar, A. Khuntia, K. Dash and S. Durani
   Journal of Radioanalytical and Nuclear Chemistry, September 2024. https://doi.org/10.1007/s10967-024-09771-5.
2. Certified reference material (CRM) of tea powder (BARC - D3201) for K, Ca, P, Mg, Mn, Al, Fe, Ba, Zn, Cu, Sr, Pb, As, Cd, and Hg: Method validation and its production
   L. Rastogi, A. Durga Prasad, D. Saikrishna, S. Yadlapalli and K. Dash
   Food Control, 158 (2024) 110241.
3. Setting up of zero-gap electrochemical prototype plant for total dissolved solids (TDS) removal from groundwater
   S. Thangavel, G. Venkateswarlu, A. Durga Prasad, L. Rastogi and K. Dash
   Lecture Notes in Civil Engineering, Vol. 581, Recent Technologies and Challenges in Water Remediation, Springer Nature (in press).
4. Development of a novel digestion method utilizing a new PTFE digestor for the rapid and reliable determination of technology-critical elements (TCEs) in granite samples by ICP-OES: A simple approach for the analysis of complex geological matrices
   M. V. Balarama Krishna, K. Chandrasekaran and G. Venkateswarlu
   Talanta, 285 (2025) 127426.

5. Development of a simple and efficient two-step microwave-assisted digestion method for the determination of REEs, HFSEs and other elements in granite samples by ICP-OES
   K. Chandrasekaran, M. V. Balarama Krishna, G. Venkateswarlu, J. George and B. Sunil Kumar
   Journal of Analytical Atomic Spectrometry, 39 (2024) 2116-2128.
6. Development of an expeditious and sensitive micro-precipitation approach using chrome azurol S and CTAB for naked-eye detection of Fe(II) and Fe (III) in waters
   M. V. Balarama Krishna, Aruna Jyothi Kora and K. Chandrasekaran
   Journal of Analytical Chemistry, 79 (2024) 773–783.
7. Renewable, natural, traditional dish wash cleaning materials used in India: An overview
   A. J. Kora
   Bulletin of National Research Centre, 48 (2024) 28.
8. Hydrophobicity induced graphene oxide based dispersive solid phase microextraction of strontium from seawater and groundwater prior to GFAAS determination
   M. Sebastian, D. Saikrishna, S. K. Jayabun, N. N. Meeravali, R. Shekhar and A. C. Sahayam
   Journal of Analytical Atomic Spectrometry, *39* (12) **(**2024) 3190-3197.
9. Interference-free ultratrace beryllium determination in alkaline effluents of beryllium processing plants by graphite furnace atomic absorption spectrometry after a novel graphene oxide-assisted dispersive micro solid phase extraction without using a chelating agent
   D. Saikrishna, N. N. Meeravali and A. C. Sahayam
   Journal of Analytical Atomic Spectrometry*, 39* (5) (2024) 1322-1331.
10. Improvement in the sensitivity of orthophosphate determination by controlling the self-reduction of excess ammonium molybdate followed by room temperature non-ionic mixed micelle cloud point extraction of anionic phosphomolybdenum blue for spectrophotometric determination
    D. Saikrishna, G. Venkateshwarlu, N. N. Meeravali and A. C. Sahayam
    Microchemical Journal, 201 (2024) 110531.
11. *In-situ* Ti-Ir and ammonium thiocyanate modifiers for improvement of sensitivity of Sc to sub parts per billion levels and its accurate quantification in coal fly ash and red mud by GFAAS
    K. Madhavi, R. Shekhar, G. Venkateshwarlu and K. Dash
    Talanta, in press.
12. Chemical characterisation of lithium-based ceramics utilizing charged particle activation and ion beam techniques

S. Dasgupta, J. Datta, G. L. N. Reddy, M. Ghosh, and K. K. Swain
Journal of Radioanalytical and Nuclear Chemistry, 333 (2024) 4919–4925.

13. High-temperature corrosion of nanocrystalline Ni with varying grain sizes in FLiNaK salt and corrosion-induced surface faceting
S. Julie, Ch. Jagadeeswara Rao, C. David, R. Kumar, K. M. Kishore, S. Chinnathambi, P. W. Nitin, A. A. Sukumar and K. S. Krishna
Surfaces and Interfaces, 53 (2024) 105052.

14. Characterization of oxides formed on Zr-2.5 (wt. %) Nb alloy in Water and Steam Environments
A. Samanta, S. Banerjee, R. Rekha, V. Srikanth, Y. Sunitha, S. Kaity, J. V. Ramana and A. K. Tyagi
Journal of Nuclear Materials, 605 (2025) 155546.

8.3. Conference presentations

1. Development of a method for the analysis of sulfur in high purity quartz using ion-chromatography and its validation by L. Rastogi, A. Durga Prasad, S. Thangavel and K. Dash, Current Trends in Analytical Chemistry, CTAC-2023,' organized by the Analytical Chemistry Division, Bhabha Atomic Research Centre, Mumbai, India, held during 6th-9th, March 2024.
2. Preparation of Dolomite Certified Reference Material (CRM BARC B1101) by G. Venkateswarlu and A. C. Sahayam, Current Trends in Analytical Chemistry, CTAC-2023, organized by the Analytical Chemistry Division, Bhabha Atomic Research Centre, Mumbai, India, held during 6th-9th, March 2024.
3. A novel sample digestion approach for Ferrocarbonatite CRM preparation for Rare Earth Elements by S. Thangavel, G. Venkateswarlu, A. Durga Prasad, K. Dash, S. Durani and B. Sunil Kumar, Current Trends in Analytical Chemistry, CTAC-2023, organized by the Analytical Chemistry Division, Bhabha Atomic Research Centre, Mumbai, India, held during 6th-9th, March 2024.
4. The Certification of Mass fractions of Na_2O, CaO, TiO_2, V_2O_5, Fe_2O_3 and Ga_2O_3 in the Alumina (BARCB1301) by L. Rastogi, A. Durga Prasad, S. P. Mohapatra, A. Khuntia, L. Naresh and K. Dash, International Symposium on Challenges and Advancement in Materials Characterization, November 20th–22nd, 2024, AMD Hyderabad.
5. Development of a hotplate digestion method using an in-house PTFE digestor for the rapid and efficient dissolution of spodumene for the determination of Li, Al, Fe and Na by ICP-OES by K. Chandrasekaran, M. V. Balarama Krishna, G. Venkataeswarlu and K. Dash, in AMD-INS International Symposium on "Challenges and Advancements in Material Characterization (CAMC-2024)", at AMDER Hyderabad, on 20th -22nd, November, 2024.

6. Application of tree gum synthesized biogenic silver and palladium nanoparticles as catalysts towards dye decolourization by A. J. Kora, in 4th International Conference on Nanomaterials & technologies (CNT-2024) at Vidya Jyothi Institute of Technology, Hyderabad on 2nd & 3rd Feb 2024.
7. Application of tree gum capped zinc oxide nanoparticles towards disinfection of wastewater bacteria by A. J. Kora. in 1st DAE-BRNS International Conference on Recent Developments in Biofilms and Biofouling Control (BBC-2024) at WSCD, BARC, Kalpakkam on 12th-14th December, 2024.
8. Development of facile, green chemistry based synthetic protocols for nanoparticles (NP), Materials Characterization-Quality Assurance in Analytical Measurements (MCQAAM-2024) by A. J. Kora and L. Rastogi at National Centre for Compositional Characterisation of Materials (NCCCM), Hyderabad, 13th Sep 2024.
9. Determination of cesium (Cs) and rubidium (Rb) in spodumene using graphite furnace atomic absorption spectrometry by Vatsal Trivedi, D. Saikrishna, K. Chandrasekharan, R. Shekhar and K. Dash, AMD-INS International symposium on challenges and advancements in material characterization (CAMC) – 2024 held at Atomic Minerals Directorate for Exploration and Research (AMD), Hyderabad, 20-22rd November 2024.
10. Determination of isotope ratio of lithium in spodumene by glow discharge quadrupole mass spectrometry by D. Saikrishna, R. Shekhar and K. Dash, AMD-INS International symposium on challenges and advancements in material characterization (CAMC) – 2024 held at Atomic Minerals Directorate for Exploration and Research (AMD), Hyderabad, 20-22rd November 2024.
11. Graphene oxide mediated extraction of strontium at ultratrace levels from seawater for its determination by GFAAS by D. Sai Krishna, S. Mariya, N. N. Meeravali, R. Shekhar and A. C. Sahayam, 3rd DAE-BRNS Symposium on Current Trends in Analytical Chemistry (CTAC–2023), on March 6-9, 2024 at DAE Convention Centre, BARC, Mumbai.
12. Studies on the extraction of lithium from seawater using leaf extract for its determination by GFAAS by Vatsal Trivedi, D. Saikrishna, S. Mariya, N. N. Meeravali, R. Shekhar and A. C. Sahayam. 3rd DAE-BRNS Symposium on Current Trends in Analytical Chemistry (CTAC–2023), on March 06-09, 2024 at DAE Convention Centre, BARC, Mumbai.
13. Methodology for high resolution depth profiling of nickel using ^{58}Ni(p, γ)^{59}Cu nuclear reaction**,** A. A. Sukumar, D. V. Lakshmipathy, G. L. N. Reddy and Y. Sunitha, Symposium on Current Trends in Analytical Chemistry (CTAC-2023), March 6-9, 2024, Mumbai.

14. Investigations on the applicability of proton induced γ-ray emission in the determination of lanthanides and heavy metals, Y. Sunitha, A. A. Sukumar, S. K. Jayabun and J. V. Ramana, DAE-BRNS Theme Meeting on "Nuclear Probes for Materials, Medicine and Industry (NPMMI-2024)" organized by BARC at DAE Convention Centre, Mumbai during June 7-8, 2024.
15. Indigenous preparation of cellulose nitrate films and their application to the autoradiographic studies of rock samples, P. Nagabhushanam, S. Bhagart, L. Hanuma Reddy, B. Sunilkumar, T. S. Sunil Kumar, S. Durani and A. A. Sukumar, at International symposium on Challenges and Advancements in Material Characterisation (CAMC-2024), 20 – 22, November, 2024, Hyderabad.
16. Carbon Induced gamma ray spectroscopy for lithium isotopic abundance determination, A. A. Sukumar, Y. Sunitha, G. L. N. Reddy, D. V. Lakshmipathy and J. V. Ramana, at International Symposium on Challenges and Advancements in Material Characterisation (CAMC-2024), 20 – 22, November, 2024, Hyderabad.

8.3.1. Best posters awards of conference presentations

1. Homogeneity assessment of candidate alumina certified reference material for Na_2O, MgO, CaO, TiO_2, V_2O_5, MnO, Fe_2O_3, Ga_2O_3 by A. Durga Prasad, L. Rastogi, S. P. Mahopatra, A. Kuntia, L. Naresh and K. Dash, Current Trends in Analytical Chemistry, CTAC-2023, organized by the Analytical Chemistry Division, Bhabha Atomic Research Centre, Mumbai, India, held during 6th-9th, March 2024.
2. A simplified method for the determination of total BF_3 in CaF_2:BF_3 complex by its extraction inorganic Lewis base by A. Durga Prasad, S. Thangavel, L. Rastogi and K. Dash. International Symposium on Challenges and Advancement in Materials Characterization. November 20th-22nd, 2024, AMD Hyderabad.

8.4. Book chapters

1. A. J. Kora, K. Madhavi and K. Dash, Analysis of materials used in nuclear reactors, book chapter in "Handbook of Materials Science, Volume V: Nuclear materials" Edited by R. S. Ningthoujam, A. K. Tyagi, Springer Nature.
2. A. J. Kora, Applications of electrothermal atomic absorption spectrometry (ET-AAS) towards various metal analyses in diverse biological and environmental matrices, book chapter 22 in "Bioanalytical Techniques: Principles and Applications", Edited by Inamuddin, Tariq Altalhi, Naif Ahmed Alshehri, Jorddy Neves Cruz, Wiley-Scrivener, ISBN: 1394314108.

8.5. Awards and honours

1. Dr. A. J. Kora name was featured in 2024 list of "Elsevier/Stanford Top 2% Researchers" from India under Nanoscience & Nanotechnology category.
2. Dr. A. J. Kora received the "ISAS Dr. Prafulla Chandra Ray Award 2024" at International Analytical Science Congress 2025, held at MS University of Baroda, Vadodara.
3. The scientific staffs of NCCCM have obtained the life membership of the Society for Materials Chemistry (SMC), India.

8.6. Other activities

1. The scientific staffs at NCCCM have served as journal reviewers for publishers of Elsevier, Springer, Springer Open, TF, Degruyter, RSC etc.
2. Dr. A. J. Kora, Dr. N. N. Meeravali, and Dr. Y. Sunitha attended orientation programme on DAE observer duties for OCES/DGFS-2024 exam on 6th March, 2024 and served as a DAE observers for OCES/DGFS-2024 exam from 15th-17th March, 2024 at Vijayawada.
3. Most of the employees of NCCCM attended DAE Vigilance Conclave 2024 @ Hyderabad jointly organized by all the units of Department of Atomic Energy located at Hyderabad on 25th October 2024 at NFC, Hyderabad.
4. All employees of NCCCM, participated in refresher lecture and mock drill on fire emergency preparedness organized by NCCCM in collaboration with Fire Services, NFC on 17th December 2024.
5. All employees of NCCCM, participated in *Swachta Pakhwada* plantation drive conducted at NCCCM.

8.7. Delivered an Invited talk

1. Dr. K. Dash delivered an invited talk on "Research and Production of Quality Control Materials (RM/CRMs) in BARC: a journey since last four decades" Symposium on Current Trends in Analytical Chemistry (CTAC-2023), March 6-9, 2024, Mumbai.
2. Dr. G. L. N. Reddy delivered an invited talk on "Ion Beam Analysis for Material Characterization" at National Conference on Frontiers of Ion Beam Science (FIBS-2024), held during 4th -7th November 2024 at Institute of Physics, Bhubaneswar IOP.

8.8. Training programme

Training on Rutherford backscattering spectrometry (RBS)/Elastic Recoil Detection Analysis (ERDA) techniques was imparted to the officers from RCD/BARC. As part of training, the principles, experimental intricacies, simulation software pertaining to RBS/ERDA were taught.

Natural landscape at NCCCM - BARC, Hyderabad