

Analytical Chemistry in Atomic Energy Programme

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Preamble:

Analytical Chemistry is an integral part of all the branches of science like chemistry, physics, biology, geology, materials science, nuclear science and technology, medicine, environment and industry. Analytical measurements are aimed at obtaining qualitative and quantitative information about the composition and structure of various materials that have relevance to both fundamental understanding as well as applications towards improving the quality of life. Analytical chemistry is of interdisciplinary nature and often called central science that contributes to every branch of science. The analytical chemistry work started in atomic energy programme during the time of Dr. Homi J. Bhabha in early 1950s with the chemical analysis of rocks and minerals for uranium and other constituents. With the advancement in the atomic energy programme, new materials were developed and deployed for various applications. The analytical work at Chemistry Group (CG), Bhabha Atomic Research Centre (BARC) has been extended to each and every branch of atomic energy programme. Apart from analytical services, methodologies were optimized / developed to cater the needs of various programmes.

Front end of nuclear fuel cycle and birth of analytical chemistry programme:

The front end of the nuclear fuel cycle consists of steps like mining, milling and leaching of uranium in rocks to get yellow cake consisting of uranium along with other impurities. Fabrication of nuclear fuel either as uranium metal for research reactors or uranium oxide (UO_2) for power reactors is the next step after purification of yellow cake from undesirable impurities. In addition to uranium and thorium, other metals like zirconium, beryllium, niobium and tantalum are also required for other reactor applications. This requires extensive work on exploration of atomic minerals with active involvement of geologists and chemists. Hence Analytical Chemistry programme was initiated by Dr. Homi J. Bhabha in early 1950s i.e. even before the formation of Atomic Energy Establishment Trombay (AEET).

Need for analytical chemistry in atomic energy programme:

For initiating a comprehensive programme on the utilization of atomic energy, it was essential to carry out a survey of rocks and minerals for the identification of areas where uranium is available at a commercially exploitable scale. Therefore, there was a need of suitable techniques for the analysis of uranium and other important metals like zirconium, niobium, beryllium etc. Analysis of uranium in water and other streams was also required for easy identification of uranium occurring areas. Since India has moderate uranium reserves, its recovery from low level sources like wet phosphoric acid or even sea water was also envisaged. However, India has abundant resources of beach sand at Kerala (and also in Odisha), which contains important compounds like zirconia, thoria, rare earth oxides, etc. Development of analytical techniques for mineral beneficiation was essential at the initial stage of atomic energy programme. Techniques for the purification and assay of uranium was also needed as the nuclear materials have to satisfy stringent quality requirements with respect to critical impurities like boron, cadmium, rare earth elements as well as other constituents like carbon, iron, aluminum etc. Stringent quality control requirements were also mandatory for other components like cladding materials and coolants. The analytical chemistry laboratory was set up to address all these requirements.

Analytical chemistry work in the formative years:

The Analytical Division, as it was called at that time, was staffed by personnel handpicked by Dr. Bhabha based on their experience and they worked in makeshift laboratories at Cadell Road, Prabhadevi even before the laboratories came up at Trombay. Both Chemistry and Analysis laboratories were located in the same premises and active interaction existed between them. The main mandate of Analytical Division was the chemical analysis of materials such as rocks and minerals for uranium and other constituents. Analysis of beryl (1955), rock samples from Jaduguda etc formed the major part of the work. Simultaneously, other materials like metals and alloys, solution samples and other materials like boron carbide for boron; uranium metal and oxide for carbon, nitrogen, and other constituents; steel samples for carbon, sulphur, phosphorus, silica as well as other critical constituents like chromium, nickel, manganese and molybdenum, were analyzed on a regular basis. The need for other techniques was felt and instruments were indigenously fabricated as well as methodologies were developed for carbon in uranium by low pressure method, analysis of helium in cover gas by gas chromatography, estimation of boron at trace levels by spectrophotometry, analysis of uranium at trace levels by pellet fluorimetry, analysis of ammoniacal and nitrate nitrogen by Kjeldahl's method, estimation of water in organic solvents by Karl Fisher method etc. At the same time, research on establishment and standardization of other techniques like neutron activation analysis (NAA), electrochemical techniques (especially conductivity, potentiometry, polarography, voltammetry, electro gravimetry), separation techniques like gas chromatography, ion exchange and solvent extraction, techniques for surface area and particle size, mass spectrometry using spark source mass spectrometer, thermal analysis techniques like home made instrument of differential thermal analysis (DTA) and thermogravimetry (TG) etc. were undertaken. Many of these instruments were quickly employed for analysis. The newly created Division in 1956 was headed by Dr. V.T. Athawale, who was a renowned analytical chemist and a strict

disciplinarian. There were experts on mineral analysis having analytical experience at Indian Rare Earths, neutron activation analysis, electroanalytical techniques, thermal methods etc. Many of them have contributed substantially to the analytical chemistry activities including training of new staff members or trainees of BARC Training School. In view of their expertise, senior staff members were nominated as members of various technical committees of Indian Standards Institution (Presently Bureau of Indian Standards). They contributed considerably for the formation of standards for iron and steel, copper and its alloys, water and effluent water etc.

The development stage of analytical chemistry programme:

The major analytical activities at the initial stage was the analysis of rocks and minerals and the vast majority of the analysis was carried out using conventional wet chemical methods (Classical techniques) after dissolution of the sample by acid digestion or fusion with reagents like sodium peroxide, KHSO_4 or by sodium carbonate. Sufficient care was taken to ensure the quality of results starting from proper coding and identification of samples, examination of the sample for its physical integrity especially in the case of weathered or moisture sensitive samples, proper sampling of the rock samples by grinding, use of certified reference materials (CRM) wherever available and use of control samples for quality assurance. The concept of accreditation of the laboratory was not in vogue at that time but the analytical chemistry laboratory was following all the requirements in an intuitive manner. Proper laboratory record books were maintained. Essential reference books were available and proper procedures were followed for the communication of results after verification of the data by the concerned supervisor. The analysis results were communicated only through the authority and were checked before dispatch by the concerned officials to avoid any typographical error. If there was any doubt on the values obtained, reanalysis was carried out using alternate techniques or through other experienced staff member. It is probably through these practices that the analytical chemistry activity was able to establish credibility and Analytical Chemistry Division (ACD), Chemistry Group (CG) is considered as the nodal laboratory of Department of Atomic Energy (DAE).

In spite of considerable analytical workload, many instruments were not available during that time. The workhorse of the ACD was a double beam spectrophotometer (early 1960s). The staff members had to schedule their analysis work for the determination of iron, aluminum, titanium, thorium, uranium etc depending on the availability of the instrument since the color formed was not stable for longer time. Instruments for the analysis of carbon and sulphur were home made. A gas chromatograph was indigenously assembled and used for analysis of helium cover gas or for dissolved gases in transformer oil. Electrochemical instruments for conductivity, polarography, electro gravimetry, voltammetry etc. were assembled and used for analysis of copper, nickel and molybdenum in rock samples and later for leach liquor samples. Instruments for determination of nitrate nitrogen by Kjeldahl method and for the determination of water in organic solvents by Karl Fisher titration were assembled and employed extensively for a large number of samples. Special fume hoods were employed to avoid contamination from acid fumes from the neighboring laboratories. Work on sodium chemistry was also initiated. In spite of lack of sophisticated instruments, good quality research was conducted on potentiometry, polarography and voltammetry.

Emphasis was given on the various analytical method development and research activities using techniques like spectrophotometry, potentiometry, electrochemical and thermal methods in addition to neutron activation analysis.

One of the most important analytical instruments procured in ACD was an atomic absorption spectrometer (AAS). It greatly simplified the analysis of solution samples and reduced the load on colorimetric determination. It was very extensively used in mineral analysis section. Other instruments for neutron activation analysis like single channel and 20-channel gamma spectrometer and proportional counters were procured or assembled. A collaborative programme on the use of NAA for forensic applications was initiated in 1969 and a multi channel analyzer based on high purity Ge-Li detector was procured as part of this programme and was employed very extensively for the analysis of forensic as well as other geological and biological samples. Collaborative programme was also initiated with Geological Survey of India (GSI) for the simultaneous analysis of rock samples for precious metals. Geological samples from IIT, Bombay and other Universities were also analyzed especially for rare earth elements which are difficult to be analyzed by other methods. An instrument for the dating of rocks by Argon - Argon method using a homemade mass spectrometer was set up and employed for the geo-chronology of rocks from various regions of India. Nuclear dating methods based on uranium - lead analysis and fission track analysis were initiated. Lunar samples made available to BARC (1974) were also analyzed by NAA. An instrument for the analysis of uranium isotopes by delayed fission neutron activation analysis (1974) was set up and utilized very extensively. The collaboration with Central Forensic Science Laboratory (CFSL) was particularly strong and led to the establishment of NAA Unit of CFSL at ACD in early 1970s. GSI was assisted to set up a laboratory at Pune and arrangements were made for the regular irradiation of rock samples at Apsara Reactor and measurement of short-lived and long-lived activities at Pune. An instrument for delayed fission neutron activation analysis was set up first at CIRUS and later at DHRUVA Reactor in late 1980s. The method involved irradiation of a uranium containing rock in the pneumatic carrier facility where the sample placed in a polymeric container (rabbit) is transported to the core of the reactor, irradiated for a fixed time, usually a minute and transported back to a neutron detector. This produces prompt neutrons which decay very fast and relatively longer lived fission neutrons from ^{235}U isotope. ^{238}U and other elements present in the sample do not undergo fission under these conditions and hence the method could differentiate between natural and enriched or depleted uranium. The technique is very fast and a large number of samples were analyzed for AMD. The uranium content could also be estimated, if the isotopic composition of uranium was unchanged.

A facility was also made available for use in collaboration with Radiochemistry Division, BARC for prompt gamma neutron activation analysis at Dhruva Reactor. The system was very good for the analysis of boron and other light elements, which were difficult to be analyzed by conventional NAA. Since the prompt gamma spectrum of isotopes is different, isotopic analysis was possible.

During that time, LECO analyzers for carbon, hydrogen, nitrogen and oxygen in metals were procured along with a CHN analyzer for organic compounds. An instrument for the determination of sulphur in steel samples was assembled. Instruments for particle size and

surface area analysis was procured or assembled. These instruments made the analysis for these requirements fast and were extensively employed.

Facilities were set up for atomic absorption analysis using graphite furnace atomizer (GFAAS) as well as electrochemical techniques and they continued to render analytical services to users. At this time, there was a requirement for the analysis of high purity materials, typically 99.999% pure (referred to as 5N pure) or better. This requirement was initially for semiconductor materials like silicon and later for III-V semiconductors like gallium arsenide. There was also a commercial angle for this analysis since the cost of high purity materials varies substantially with its purity. In the absence of a suitable analytical laboratory, the country had to depend on the certificate given by the manufacturer. There was also a programme for the production of high purity gallium and arsenic in the Chemistry Group in addition to the preparation of other metals in a highly pure form in collaboration with Nuclear Fuel Complex (NFC), Hyderabad. The preparation of such materials considerably depended on the availability of reliable analytical data so that the preparation route can be standardized in addition to ensuring the purity of final product. In view of very low levels of impurities often present at parts per billion levels, considerable effort is required for the purification of reagents and chemicals, especially acids, water and other solvents, vessels used for sample dissolution and selection of sensitive and selective analytical technique. A laboratory was set up in early 1980s for this purpose (Ultra Trace Analytical Facility: UTAF) at Training School complex (South site), BARC so that the contamination due to laboratory environment and unauthorized entry of personnel can be avoided. The air entering the laboratory was purified using HEPA filters and laminar flow work benches (Class 100) were employed. The materials used in the laboratory, the work benches and even other facilities like laboratory sinks, taps etc had to be made of plastic to avoid metal contamination. Dedicated instruments like GFAAS, electroanalytical analyzer were employed. Facilities were developed for the purification of acids by sub-boiling method.

A laboratory was set up at Variable Energy Cyclotron Centre (VECC), Kolkata in early 1980s for the development of analytical facilities using heavy/light ion beam available at VECC. Charged particle activation analysis (CPAA) was a complementary technique to NAA and enabled the analysis of elements not so sensitive by NAA. Thin layer activation (TLA) and ion implantation techniques were also developed in the laboratory at VECC at later stage.

Analytical chemistry in 1990s and augmentation of analytical facilities:

Although ACD was rendering analytical services and carrying out research work on solution chemistry, potentiometry, solvent extraction and ion exchange, electrochemistry and thermal analysis, there were difficulties due to ageing of instruments. Many of the instruments had become outdated and suffered from frequent breakdown. Therefore, new procedures were developed using suitable separation methods and using available instruments. In spite of non-availability of sophisticated instruments, ACD, CG continued its participation in collaborative projects with other National Agencies like CFSL, GSI as well as with Hindustan Zinc Ltd, Heavy Water Board (HWB), Clean Ganga Project etc. This enabled the ACD to obtain ion chromatograph for the determination of chloride in heavy water at ultra trace levels. During this time, use of direct current plasma (DCP) emission spectrometer enabled the analysis to be completed in a matter of minutes and large numbers of samples

were analyzed. DCPAES was obtained as part of Clean Ganga project. A thermal analyzer, DTA-TG-MS was also obtained and commissioned. This instrument enabled the thermal characterization of many materials and helped in the optimization of sintering procedure for uranium oxide produced in NFC. Similarly, a homemade sulphur analyzer was employed very extensively when there were problems due to cracking of thoria pellets produced at NFC. The studies enabled an analysis of root cause for the problem and standardization of newer procedure to minimize the difficulties.

The augmentation of analytical chemistry research facilities came through the efforts of officials of ACD in 1995. Some of the instruments procured were AAS, ICPOES, carbon-sulphur analyzer, high performance liquid chromatography (HPLC), ion chromatograph (IC), inductively coupled plasma mass spectrometer (ICPMS), spectrofluorimeter, electrochemical instruments, gas chromatograph (GC), particle size analyzer, elemental analyzer etc. This substantially increased the instrumental analytical capabilities of the Division.

With the increasing demand for characterization of high purity and other advanced materials, National Centre for Compositional Characterization of Materials (NCCCM), CG was set up at Hyderabad in 1993 with the efforts of Dr. S. Gangadharan. The centre is well-equipped for analysis of high purity materials as well as for ultra trace level impurity analysis in various technologically important materials (Ultra Trace Analytical System: UTAS). Both ACD and newly created NCCCM also participated in inter comparison exercises on a regular basis.

Renaissance in analytical chemistry:

The later period saw an increase in other activities like participation in inter comparison exercises conducted by IAEA, NPL, GSI etc and those initiated by DAE. Utilization of instruments was increased and training programmes were also conducted. A large number of samples were analyzed for ONGC by GFAAS as part of their work on monitoring of metallic contaminants in sea around the regions of offshore drilling. Analysis of boron isotopes was carried out for a DAE project. ACD participated in the work on the preparation and standardization of analysis procedures for trace metallic impurities in uranium oxide, analysis of hydrogen in zircaloy tubes, preparation of standards for boron and boron carbide etc. Training was imparted to a large number of students from Mumbai and other Universities for their M.Sc. project work. Design and fabrication of some instruments were also started due to nonavailability of instruments or service support from other countries. Development of electrochemical instruments and development of computer software for online processing of analysis requests were made. Various electrochemical sensors were developed for detection of toxic elements as well as biomolecules. Various sorbents were synthesized and applied for removal of toxic elements / radionuclides from various systems.

ACD had earlier participated in the development of a mercury analyzer for the determination of mercury in fish. A test kit for the spot test of metals in steel samples was developed. X-ray fluorescence (XRF) instrument was set up which enabled rapid non-destructive testing of many materials including metals and alloys, precious metals like iridium and jewelry materials. The rapid non-destructive analysis of gold ornaments fascinated many students visiting the facility. Spark source optical emission spectrometry

(Spark-OES) enhanced the non-destructive as well as rapid analysis capability for composition of Fe/Al/Cu based alloys. X-ray diffraction (XRD), scanning electron microscope-energy dispersive spectrometry (SEM-EDS), differential scanning calorimetry (DSC), atomic force microscopy (AFM) etc. enhanced the analytical capabilities.

Tandemtron accelerator facility was developed in NCCCM in 1995. Methodologies were developed and deployed for surface elemental composition as well as depth profiling of impurities in various technological important materials using Rutherford back scattering (RBS), nuclear reaction analysis (NRA), particle induced X-ray emission (PIXE) and particle induced gamma ray emission (PIGE) techniques using this accelerator facility.

With the changing scenario over the world, the need was also felt for obtaining formal recognition for the analytical laboratories for international acceptance of analytical data generated. Both NCCCM and ACD pursued this activity and both the laboratories obtained accreditation as per ISO 17025 from National Accreditation Board for Testing and Calibration Laboratories (NABL) in 2009 and 2019, respectively. NCCCM, CG also obtained NABL accreditation as per ISO 17034:2016 for the production of certified reference materials (CRMs) in 2018.

Indigenous development of analytical instruments is another important field and under this programme, carbon analyzer for uranium samples, carbon-sulphur (C/S) analyser, surface area analyser, improved hydrogen determinator, dissolved oxygen monitors were developed and are currently used for various analyses.

An important area was the study of spectroscopic properties of ^{233}U , which was obtained from irradiated thorium through protactinium (Pa). For studying the behavior of Pa, a method was developed for the separation of ^{231}Pa from various materials including natural sources such as siliceous cake. Other requirements where analytical chemistry played a major role were: speciation of methyl mercury by gas chromatography, removal of arsenic from ground water, trace element distribution of heavy metals in algae as a method of monitoring environmental pollution, distribution of metal ions in food grain or in opium as a method of correlating trace element profile with the region where they were grown. Various portable kits were developed for field detection of toxic components such as fluoride / arsenic / chromium in ground water and deployed. The technology for development of these kits has been transferred to several private companies for its commercialization.

Few of the important areas in atomic energy programme where CG, BARC has provided analytical solutions recently (2020s) are discussed in this section. Impurities determination in uranium metal for research reactors; composition / impurities of nuclear reactor structural materials / claddings; development of new shielding materials for Indian reactors; ceramic magnets used in reactor control system; blood/serum/plasma samples from patients of ACTREC/TMH for target element determination; deuterated liquid scintillator (DLS) development for neutrino detection programme; composition of oxygen and other gas samples from electrolysis plants/ N_2 plant; AGMS CO_2 and O_2 gas from power reactors; KKNPP turbine oil for solving frothing problem; samples from BRIT for radiopharmaceutical preparation; sample from Lu enrichment programme for radiopharmaceutical preparation; REE oxides from IREL; Al samples for aerospace application; toxic elements in sludge samples from SHRI facility, high temperature Ni/Ti alloys for fusion reactor programme; serpentine ore pebbles for shielding purposes; materials from LINAC; Sm-Co /Nd based

materials for permanent magnets development; Li based ceramics; chickpea/rice/wheat samples for micronutrient studies; protein / plant extracts; process samples for laser isotope separation (LIS) studies on Sm/Yb/Lu, waste management study, Sr immobilization studies, corrosion study, Ga separation work at HWB, arsenic leaching studies / CFL leachates; geological soil/rock/sediments for baseline studies; water collected under Jal Shakti Abhiyan for major, minor, trace or ultra-trace constituents; groundwater samples from various places in India for source studies; Cement/blast furnace slag; Pt/Pd based catalysts for hydrogen storage/generation applications; desalination membranes development; nanoparticles for different applications; geopolymers/ glass lubricants/glass materials for waste management; organometallic development; and mice tissues for radio protector development. Analytical services is regularly being provided to the projects of national importance such as helium exploration in which large number of borehole and hot spring gases amples received from AMDER were analysed and lithium exploration for which total lithium contents as well as isotopic ratio measurements were carried out.

The analytical chemistry activities started with mostly bulk analysis (% level) using classical techniques like gravimetry and titrimery for rocks and minerals. Though the methods had good accuracy, they were time consuming and laborious. With the availability of techniques like AAS, NAA and electroanalytical techniques etc., trace level determination was possible in wide varieties of matrices. Analysis at ultra-trace concentration levels was possible with the development of UTAF/UTAS for handling high purity materials and deployment of instruments like ICPMS, ICPOES, GFAAS, and TXRF etc. Newer analytical techniques/ instruments not only have lower detection limits but the analysis times are considerably less leading to lower turnaround time. Though instrumental techniques are commonly employed now, classical analytical techniques always have their special place in analytical laboratory.

Certified/in-house reference materials production programme:

Reference materials (RM) are commonly used for validation of existing analytical methods and during development of new methodologies. The use of Certified Reference Materials (CRMs) is a widely accepted tool for verification of the accuracy of analytical methods and they form an integral part of the laboratory quality control systems. The matrix matching CRMs are very costly and also not always available. In order to overcome these issues, Chemistry Group (CG), BARC initiated a unique programme for the development of CRM/in-house reference materials in 1970s to cater to the need of DAE and non-DAE institutes as well as to satisfy needs of Indian industrial sector together with the needs of social sectors. Under this programme, various certified/in-house reference materials have been produced at CG, BARC and utilized for quality control purposes.

First in-house geological standard, TKT-1 (Trachy basalt from Kandivali, Bombay) was prepared in ACD, CG way back in 1976. Concentrations of 13 elements, Na, Mn, U, Th, Hf, Sc, La, Eu, Tb, Ce, Co, Rb, Cs were established in TKT-1. As the amount was less, it was consumed in short time. Therefore, next in-house geological reference material B-78 was prepared at ACD after collection of 25 kg of black basalt from Gilbert hill, Andheri, Bombay. The rock was broken into small pieces, passed through jaw crusher, powdered in ball mill. 3kg homogenized material was bottled and homogeneity test was carried out using ANOVA.

Concentrations of 29 elements (12 major / minor and 17 trace) were assigned using classical methods, spectrophotometry, AAS, flame emission spectrometry (FES) and INAA techniques. It was established that the material was homogeneous at 100 mg level.

In-house hair reference material for mercury was prepared at ACD in 1992. For this work, 35 kg of hair was purchased from Tirumala Tirupati Devasthanam, Tirupati. 10 kg material was processed which involved cleaning several times with acetone / deionized water, cutting into pieces, radiation sterilization with 50 kGy at ISOMED, Mumbai. Homogenization exercise was carried out at International atomic energy agency (IAEA). Mercury was estimated using cold vapor AAS and NAA. Concentrations of Se, Zn, Cd, Co were also determined.

Bharatiya Nirdeshak Dravya (BND) i.e. Indian Certified Reference Material (CRM) of high purity quartz (BND 4101.01) powder for trace elements Al, Fe, Ca, Na, K, Mg, and Ti was prepared at NCCCM, CG in 2016. About 10 kg of quartz was collected from quartz mines in Tamil Nadu. The material was milled to powder form 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. 25 g of aliquots were packed into 350 pre-cleaned high-density polyethylene (HDPE) bottles. The trace element impurities were certified by NCCCM and CSIR-NPL by means of an inter-laboratory comparison exercise (ILCE). Analytical techniques used for trace elements measurements in quartz were ICPOES, AAS and total reflection X-ray fluorescence (TXRF) spectrometry.

Graphite is extensively used in various industries and boron (B) concentration in graphite is one of the important parameters that decide its acceptability for applications in nuclear industry. Because of the importance of B concentration in graphite and unavailability of graphite reference material, an in-house graphite reference material (ACD-G-16:100 bottles of 30 g each) was prepared at ACD in 2016. ICPOES, ICPMS and CPAA techniques were utilized for assigning concentration of B. Homogeneity testing was carried out on the bottled units along with stability evaluation and boron concentration along with the combined / expanded uncertainties were established.

NCCCM, Hyderabad participated in the Inter Laboratory Comparison Exercise (ILCE) for the development of 4N pure gold certified reference standard (120 gold standards of 20 g each) named Bhartiya Nirdeshak Dravya, BND 4201.

Food Safety and Standards Authority of India, (FSSAI) has set maximum permissible level of lead (Pb) in noodle at 2.5 ppm (mg kg^{-1}). Considering the unavailability of matrix matched reference materials in India, NCCCM had produced 2.35 kg (80 bottles) of noodle CRM (BARC D3101) for Pb in 2018. The produced CRM was tested for bulk and analytical mass homogeneity. After assuring the homogeneity, Inter Laboratory Comparison Exercise (ILCE) was conducted and based on the results Pb concentration was assigned as $(2.55 \pm 0.12) \text{ mg kg}^{-1}$. The produced CRM was assessed by NABL as per ISO 34 guidelines.

Uranium metal is used as fuel in research reactor in BARC, Mumbai. Due to unavailability of uranium metal RM, in-house uranium reference material was prepared in 2018 for evaluating concentrations of Fe and Al. ICPOES technique was used for the determination of these elements after matrix separation. Environmental samples such as water and soils are routinely analyzed at ACD. Due to high cost of the commercially available

CRMs and continual use, In-house RMs were also prepared in 2019/2020/2022 for the concentration of trace elements (Fe, Zn, Cr, Mn, Co, As, Cd, Al, U) in potable water, (Al, Cr, Cu, Mn, Ni, Zn) in soil and (Co, Cr, Pb, Cu, Mn, Ni, Zn, Fe) in marine sediment. Concentrations were assigned using ICPOES and other analytical techniques. In-house seawater reference material was prepared at ACD in 2021 for evaluating the concentration of trace elements. Standard protocols were followed for seawater collection, spiking with trace elements and homogenization. Total 100 bottles, containing 200 mL of seawater were prepared. Long term stability tests were also carried out. The concentrations of Al, Cr, Cu, Mn, Zn, Ni and Cd elements were measured in solution with high salt content using ICPOES.



Fig. 1: Photographs of CRMs/in-house RMs: (i) Bauxite (BARC B1201) (ii) Dolomite (BARC B1101), (iii) Quartz (BND 4101.01), (iv) Marine sediment (ACD-MS-01), (v) Zirconium boride (BARC/QCM-ZrB₂/2023), (vi) Graphite for boron (ACD-G-16)

An indigenously developed Dolomite CRM (BARC B1101) was released on November 9, 2021 by Chairman, Atomic Energy Commission (AEC). The dolomite CRM was prepared in accordance with the ISO guidelines jointly by Atomic Minerals Directorate for Exploration and Research (AMD) and NCCCM. “Aatmanirbhar Bharat”, the concept towards self-reliance was the key motivation behind this exercise. The starting material was sourced by

AMD from Tumallapalle area, YSR district, Andhra Pradesh. The cost of indigenous dolomite CRM, (BARC B1101, 50 g packing) is about one third of the commercial ones and is easily available to users.

India's first Bauxite CRM (BARC B1201) was recently developed by NCCCM in collaboration with NALCO and released on March 24, 2023. This is the first of its kind CRM in India and 5th in the World. The CRM was certified for nine property values such as concentrations of Al_2O_3 , Fe_2O_3 , SiO_2 , TiO_2 , V_2O_5 , MnO , Cr_2O_3 , MgO and loss on ignition (LOI), which are traceable to the international system of units (SI). According to the press release by Ministry of Mines, this product will act as an import substitute for use by Industries, Research Laboratories and Academic Institutions as a calibration standard in evaluating analytical methods, performance of instruments, and data quality control in routine analysis of bauxite.

Recently in-house Reference material of zirconium diboride (ZrB_2) was prepared for boron and carbon (2023). Homogeneity assessment, stability checking and value assignment for boron and carbon was carried out.

BARC chemical incinerator:

In early 2010, Director, BARC had given the responsibility to Head, ACD, CG to look for safe disposal of legacy and unused chemicals in BARC. Various options were explored and a chemical incinerator with a capacity of 20 kg/hour for organic liquid chemical waste was installed to dispose used / legacy chemicals and operation started in 2016 (Fig 2). The incinerator is being maintained and operated by Analytical Chemistry Division (ACD), BARC. Pre-operational environmental surveillance at "BARC chemical incinerator site" was carried out. So far, more than 60,000 litres of organic liquids from 25 Divisions of BARC, were safely disposed in the chemical incinerator. Monitoring of various flue gases during the operation of chemical incinerator is carried out on regular basis using various sensors. The ash content obtained from the primary combustion chamber and liquid waste generated after treatment were continuously monitored for major and minor elements, pH and TDS.



Fig. 2: Photographs of different parts of the chemical incinerator installed at BARC (i) Main combustion chamber (MCC) and Post combustion chamber (PCC) of chemical incinerator, (ii) Feeding vessel, air compressor, spray nozzle, and (iii) Sensor for environmental monitoring

Way forward:

Analytical chemistry is a user driven science. The requirements are constantly changing and often there are requirements for rapid development of analytical techniques for a specific problem. In view of increasing concern about environment and industrial pollution, such requirements are likely to arise anytime and the analytical chemistry will play a major role in providing a suitable solution in the shortest possible time. With the vast expansion of atomic energy programme in India, newer types of nuclear reactors and accelerators are being developed in which various new materials will be deployed. Nuclear agriculture and food technologies also demand analytical methodologies for evaluation of desired developed materials. Newer analytical requirements will arise for characterization of all these materials, separation of ultra trace levels of impurities, non-destructive testing and management of waste using green technologies.

Analytical Chemistry Division (ACD), Mumbai and National Centre for Compositional Characterization of Materials (NCCCM), Hyderabad of Chemistry Group (CG), BARC are at the forefront in providing analytical solutions for changing needs of all DAE projects from its inception as well as for societal application and will always be in a position to tackle the present and future challenges in analysis.