

# 1. ACCELERATOR

## 1.1 15 UD PELLETRON ACCELERATOR

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### 1.1.1 Operational Summary

The 15 UD Pelletron accelerator performed well during 1st April 2020 to 31st March 2021. In spite of the COVID pandemic, 340 shifts of beam was delivered to around 40 users from various Universities/Institutes. Maximum terminal potential at which the beams were delivered was 13.66 MV and minimum terminal potential was 8.54 MV. Maximum terminal potential attained during conditioning (without beam) was around 14.8 MV. Figure 1 shows the voltage distribution graph of the Terminal Potential used for Beam runs for the mentioned period.

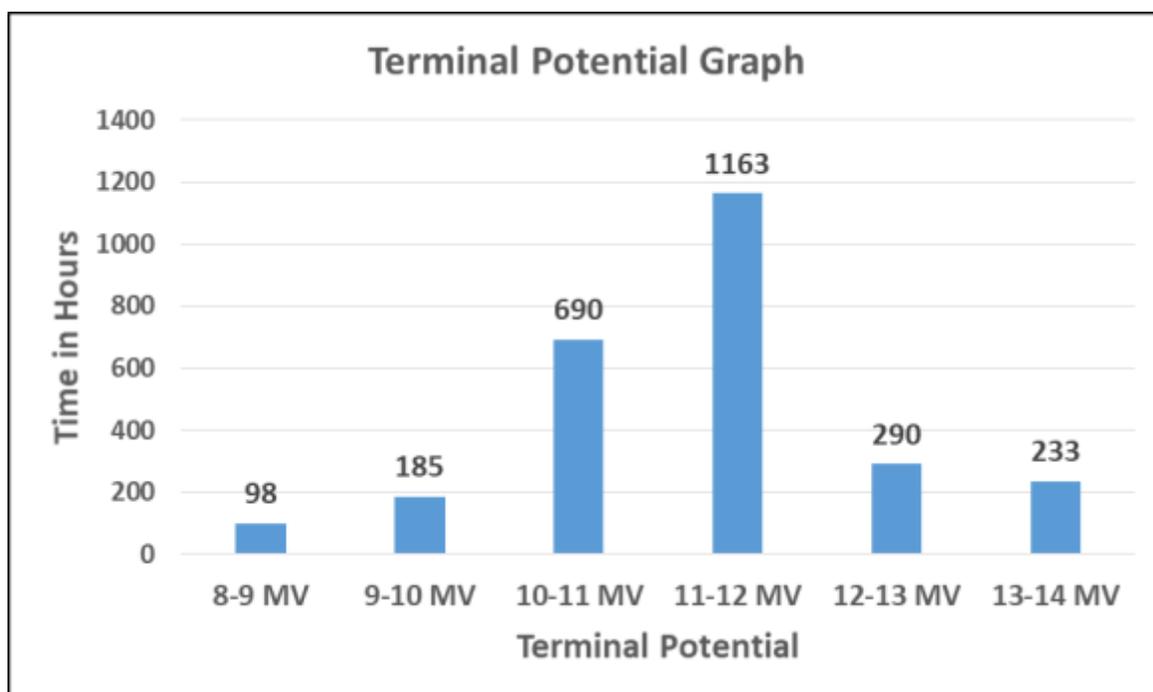


Fig 1: Voltage distribution graph in Hours.

${}^7\text{Li}$ ,  ${}^{12}\text{C}$ ,  ${}^{13}\text{C}$ ,  ${}^{14}\text{N}$ ,  ${}^{16}\text{O}$ ,  ${}^{18}\text{O}$ ,  ${}^{19}\text{F}$ ,  ${}^{28}\text{Si}$ ,  ${}^{48}\text{Ti}$ ,  ${}^{58}\text{Ni}$ ,  ${}^{107}\text{Ag}$ ,  ${}^{109}\text{Ag}$ ,  ${}^{127}\text{I}$ ,  ${}^{197}\text{Au}$  were delivered to the users for their experiments. The operational summary of the accelerator from April 2020 to March 2021 is mentioned below.

Total No. of Chain Hours	=	5060 Hours
Total Beam utilization	=	2660 Hours
Breakdown during operation	=	490 Hours
Accelerator Conditioning	=	1900 Hours

Around 60% of beamtime were utilized by the users of nuclear physics facility. Most of the nuclear physics beamtime were in HIRA followed by GPSC and INGA/HYRA. Materials science users included users from Department of Space, Bengaluru, Chandigarh and Ahmedabad. Figure 2a shows the distribution of beamtime with respect to different experimental fields and figure 2b depicts the beamline wise distribution of the beamtime.

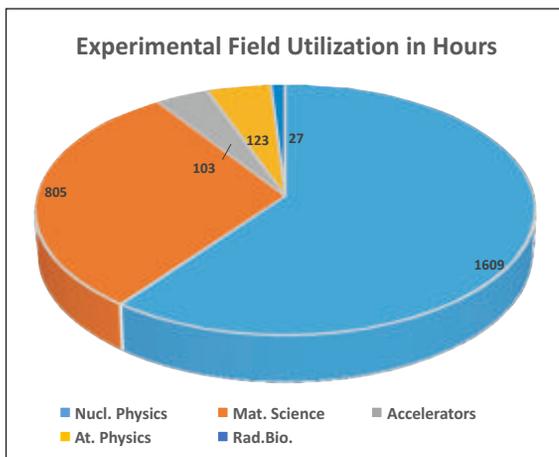


Fig 2a: Experimental field wise breakup of delivered beamtime.

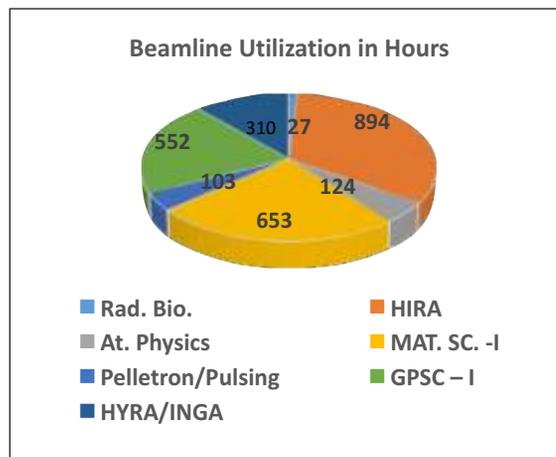


Fig 2b: Beamline utilization in hours.

The accelerator was operational with two unit shorted from month of August to December 2020 because unit number 25 and 28 were sparking continuously. Most of the experiments were conducted using gas stripper in the terminal as the strippers in the terminal and dead section 2 had gone bad due to prolonged usage. But overall the accelerator operated smoothly.

**1.1.2 Maintenance**

In the academic year two major maintenance were carried out in 15 UD Pelletron accelerator. A short breakdown maintenance was performed in the month of August and a major scheduled maintenance was performed during January – February 2021. Both the maintenances are described in detail in the following sections.

**1.1.3 Breakdown Maintenance (6<sup>th</sup> August 2020 – 11<sup>th</sup> August 2020)**

During the operation it was observed that suppressor voltage of the charging system was not going up. Ideally it should be the same as charging voltage, but it was not holding more than 2 kV. The tank was opened and it was observed that the cable connecting the high voltage feed through to the suppressor plate had got punctured (Fig.3). It was decided to just solve this particular problem as other sections of the accelerator were working fine.



Fig 2b: Beamline utilization in hours.

The tank was closed soon after replacing the cable and we could achieve 12.5 MV with 1 unit shorted. Unfortunately within a week of operation we had to short one more unit, that is unit number 25. The accelerator was operated with 2 units shorted with maximum potential reaching around 12.0 MV.

**1.1.4 Scheduled Maintenance (05/01/2021 – 20/02/2021)**

Schedule maintenance of 15 UD Pelletron was carried out during January-February 2021. Major works carried out during this maintenance are described below:

**1.1.4.1 Column Support Post Change:** The gaps of column support posts are shorted when the particular gap becomes weak electrically or mechanically. 17 gaps in low energy section and 23 gaps in high energy section were shorted. That means more than a unit was shorted in high energy section. 7 gaps in unit 28 and 3 gaps each in unit 16 and 22 were shorted in high energy section. In low energy section worst performance is of unit 7 with 3 gaps shorted. After analysis it was observed that if three posts are changed in high energy section (one each in unit 16, 22 and 28) the shorting will reduce from 23 to 14. Therefore P3 post of unit 28 and 22 and post P4 of unit 16 were changed. This reduced the number of shortings and the enhancement of potential was observed during operation.



Fig 4: Column support post replacement setup.

**1.1.4.2 Foil Stripper Replacement:** Fresh stripper foils were loaded in Terminal and Dead Section 2. In the terminal area, laser plasma ablated foils (LPA foils) procured from Technical University of Munich were loaded. LPA foils are known to possess longer lifetime compared to conventional foils fabricated in house. Therefore these foils are preferred during LINAC experiments. In-house fabricated foils were loaded in dead section 2. These foils are used to enhance the charge state of incoming beam resulting in increase of its energy.

**1.1.4.3 Ion Pump Replacement:** The vacuum inside the accelerating tubes and terminal area is maintained by the ion pumps installed at different locations viz. one each at two dead section and two ion pumps in terminal of the accelerator. During operation, it was observed that ion pump T2 had stopped working and performance of ion pump D2 deteriorated. After opening of tank it was found that both the pumps were shorted. New pumps were installed and vacuum of the order of  $10^{-8}$  torr was achieved within couple of days.

**1.1.4.4 Corona Probe Maintenance:** Needles of corona probe looked blunt. Therefore they were replaced with new needles. Appropriate care was taken to ensure that all needles have equal lengths. After replacement of needles, insulation of corona probe was measured and found to be in giga ohms.

**1.1.4.5 Mechanical System Maintenance:** During maintenance only 14 numbers of bearings and one rubber coupling (between unit 4 and 5) were replaced. Going by past records this is a very small number.

**1.1.4.6 Routine Maintenance:** Measurements of all resistors, column support post gaps, hoop screw measurements and replacements, cleaning of chains and charging systems and routine cleaning of Pelletron accelerator was performed. Earthquake protection setup was tested prior to tank opening.

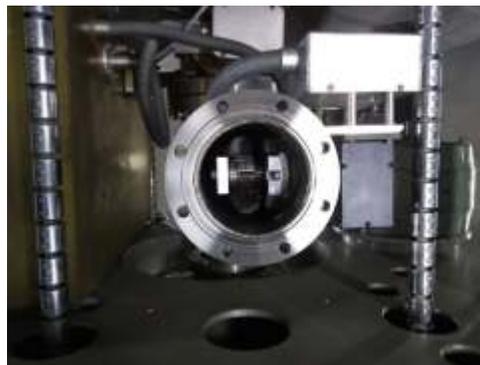


Fig 5: Foil stripper assembly of dead section D2.



Fig 6: Corona probe insulation measurement setup.

### 1.1.5 Ion Source activities

40-cathode Multi cathode SNICS was operational throughout the year without any major hiccups. Cathodes were loaded in the source on 8<sup>th</sup> June 2020, 21<sup>st</sup> August 2020, 18<sup>th</sup> November 2020, 4<sup>th</sup> December 2020 and 23<sup>rd</sup> March 2021 as per the beam schedule. No scheduled maintenance was carried out of the source. The source was cleaned thoroughly and conditioned during February 2021 after fire accident in staircase. General Purpose tube could hold 350 kV post conditioning.

#### 1.1.5.1 Replacement of Ionizer

Ionizer of the source broke down on 13<sup>th</sup> October 2020 during an experiment. This ionizer lasted for around two and half years of routine operation. The ionizer was replaced, baked, and the beam was delivered within couple of days.

### 1.1.6 Activities during lockdown

Last academic year we had to go through lockdown due to the prevailing pandemic condition. During lockdown accelerator and ion source were totally shutdown. All the vacuum pumps and power supplies were kept off. To ensure safety of the Pelletron tank, SF<sub>6</sub> pressure was reduced to 45 PSIG and for the safety of the accelerator column, earthquake protection arms were kept inserted. Group members took turns to visit and inspect the facility. The lockdown period was also utilized to collate information and prepare a manual related to the operation, maintenance, possible upgradation in the accelerator facility. The report has been submitted in the office.

## 1.2 SUPERCONDUCTING LINEAR ACCELERATOR (SC LINAC)

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During the operation of the Superconducting Linac extending from December 2019 to March 2020 in the previous academic year, it was observed that the performance of some of the QWRs, especially in the second and the third accelerating modules, was poor. The gradients achieved by these resonators are shown in red in the bar chart shown in figure 1.

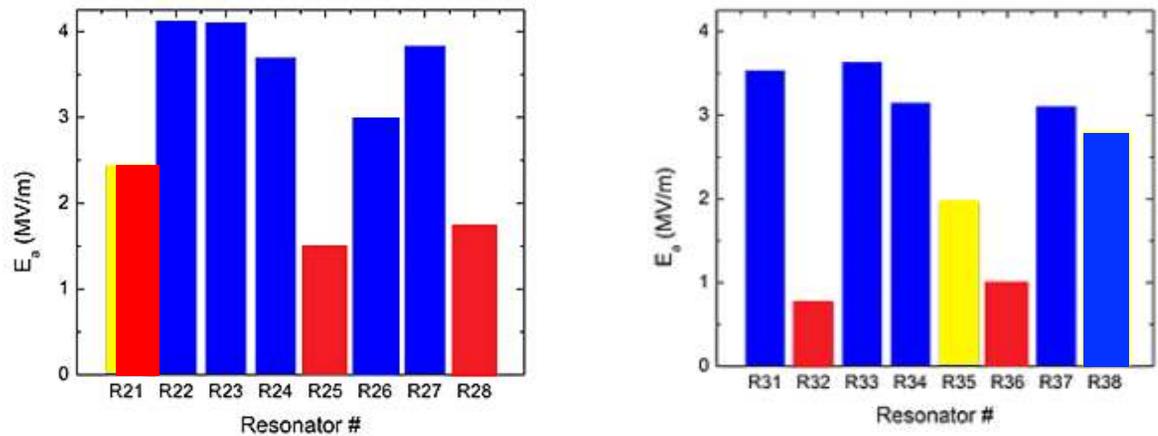


Fig 1: Accelerating gradients in 2<sup>nd</sup> and 3<sup>rd</sup> accelerating modules during linac operation. Red bars indicate resonators requiring additional surface processing.

was decided to take down these cavities after the run and give them the necessary surface treatment to improve their performance. It was also planned to align the solenoid magnets installed inside the linac cryostats to a greater accuracy and use them in future operations to improve the beam quality and transmission through the linac.

However, due to the lock down following the Covid pandemic and subsequently with the social distancing norms in place, the scheduled linac maintenance had to be postponed. So, it was decided to carry out all the aforesaid tasks in the next academic year. Meanwhile preparations are underway for another cycle of the SC Linac operation which is expected to begin shortly. These include RF and frequency checks of the cavities, testing of power coupler movements, checking the piezo and PWM tuners, RF multiplexer modules, etc.

### 1.2.1 Work done during the lock down period

During the lockdown period with work from home in effect, major documentation work was completed. Several reports related to various aspects of the SC Linac were prepared by the group members, which include:

1. A report on “Control scheme for superconducting linear accelerator at IUAC”
2. A report on “Operation of the Superconducting Linac”
3. A report on “Multipacting Basics”
4. Two reports respectively on “Analytical calculation of resonant frequency of (i) stepped and (ii) tapered transmission lines”
5. A report on “Piezo control module”
6. User manual for the Resonator Controller
7. Operational manual for IUAC EBW machine
8. A report on “VME Interface control”
9. User manual for the multiplexer control
10. A report on “Nuclear Electronics module for DAS of Linac”

Since the relaxation in the *work from home* directive, servicing of all the RF power amplifiers, resonator controllers, piezo controllers and other associated electronic modules has been carried out. The helium gas based pneumatic frequency tuner system installed in the first linac module has been thoroughly tested and the accelerator is being readied for beam acceleration.

## 1.2.2 Superconducting Niobium Resonator Fabrication

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### 1.2.2.1 Cold test of QWR # I 11

The fabrication of QWR # I 11 was completed towards the end of the previous academic year. The cavity was pressure leak-tested in the test cryostat. Thereafter, in the present academic year it was electropolished to remove  $\sim 100 \mu\text{m}$  of niobium material from the RF surface followed by ultrasonic cleaning and given a high pressure rinse. A subsequent cold test of the cavity at 4.2 K was performed in the test cryostat where it achieved an accelerating gradient  $E_a$  of 3.9 MV/m @ 4W of RF input power. The resonator was then warmed up and held in the temperature zone of 120-140 K for  $\sim 12$  hours and again cooled down to 4.2 K. Q measurements performed thereafter indicated presence of a mild 'Q disease'. A deterioration of  $\sim 20\%$  in the quality factor was observed @ 4W as compared to the initial test. The Q curves for the resonator are shown in figure 2.

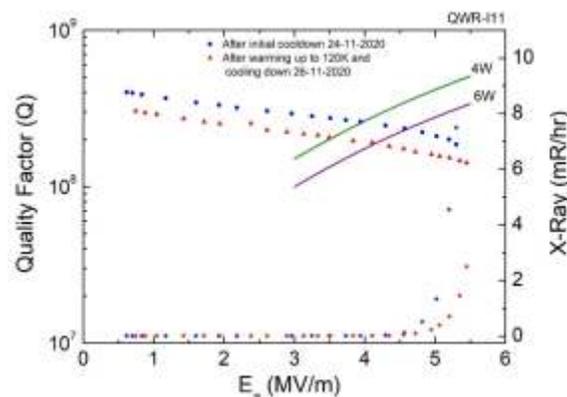


Fig 2:  $Q$ - $E_a$  curves at 4.2 K for QWR # I 11 in the baseline test.

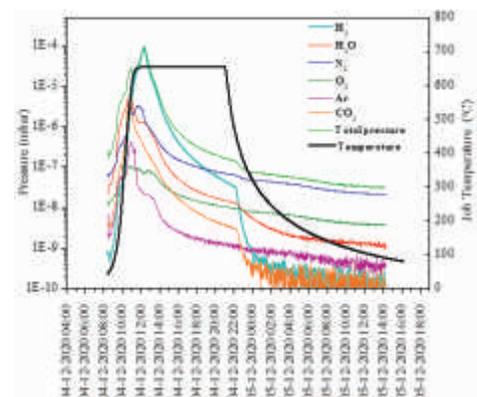


Fig 3: RGA spectrum during the 650°C heat treatment of QWR # I 11.

After the cold test, the resonator was heat treated in the high vacuum furnace @ 650°C for  $\sim 10$  hours. Figure 3 shows the RGA spectrum for the heat treatment cycle. The resonator was subsequently high pressure rinsed and loaded in the test cryostat for another test. In the second cold test performed at 4.2 K, the quality factor at low accelerating gradients (around 0.6 MV/m) showed improvement of about 50% but at higher gradients no improvement was observed (figure 4). The ultimate accelerating gradient achieved by the QWR also did not improve any further. The QWR was then warmed up to 120 K and held there for about 15 hours. After cooling it back to 4.2 K no change in its performance was observed confirming that the resonator was now free of Q-disease.

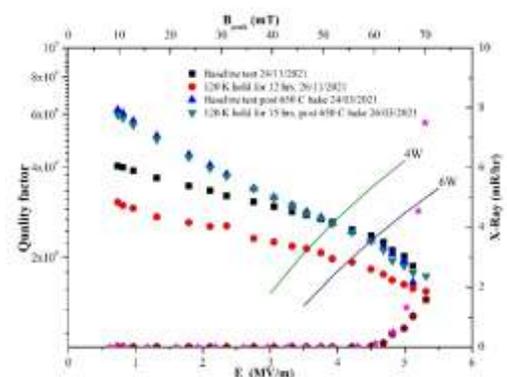


Fig 4:  $Q$ - $E_a$  curves at 4.2 K for QWR # I 11 post the 650 °C heat treatment.

### 1.2.2.2 Fabrication of six spare QWRs and fourteen spare slow tuners

All niobium components along with the outer stainless steel jackets are ready. The electron beam welding of the niobium components, however, could only be started in January this year. This is because the electron beam welding machine was non-operational for a major portion of last year due to a faulty pneumatic actuator of the high vacuum valve installed on the machine's vacuum chamber. The shipment of a spare actuator from the manufacturer, M/s Techmeta, France, was delayed due to the COVID 19 lockdowns imposed around the world. Since the machine became operational, electron beam welding of eight drift tube beam ports to their saddles; two

welds per assembly, have been completed (figure 5). The assemblies have been thereafter machined and fitted in the respective drift tubes and completion of the drift tube assemblies began (figure 6). In figure 7, a completed drift tube assembly is shown.



Fig 5: Electron beam welding of the niobium drift tube beam port with its saddle.



Fig 6: (Left) Drift Tube tacking setup, (right) Drift Tube welding setup.



Fig 7: A completed niobium Drift Tube assembly.

## 1.3 PARAS: 1.7 MV (5SDH-2) Pelletron Accelerator based Ion Beam Analysis Facility

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### 1.3.1 Operation

The 1.7 MV Pelletron accelerator for Rutherford Backscattering Spectrometry (RBS), BBS-Channeling, RBS resonance, Elastic Recoil Detection Analysis (ERDA) measurements is in continues operation. In the year 2020-21 total 1528 measurements of 28 users from 15 Universities, colleges and institutes were performed. The facility was operational throughout the year excluding the lockdown period. The Operation in this period was smooth and stable. The operational summary of 1.7 MV (5SDH-2) Pelletron Accelerator for Ion Beam Analysis activities is consolidated in table 1.

**Table 1: The Operation details of Terminal Voltage, Charge State, Energy for different measurement technique using He beam**

Terminal Potential (MV)	Charge State	Energy (MEV)	Measurement	Application
0.86- 0.987	+1	1.8-2	RBS and Channeling	Thin films, Nuclear targets Single crystals, Epitaxial layer
1.07-1.25	+2	3.045-3.09	O resonance RBS	Near and depth probe for O
1.198	+2	3.61	N resonance RBS	Near and depth probe for N
0.92	+2	2.8	ERDA	Thin and bulk targets

### 1.3.2 Maintenance

#### 1.3.2.1 Ion Source Maintenance

The low beam current from the ion source was observed in the mid of March 2020. The RF ion source had fluctuation probe current and instability of beam and decided for the ion source maintenance. The facility was shut down from March 22nd To June 10th, 2020 in lockdown due to pandemic-COVID19. The Ion source maintenance was performed during July-August 2020. The ion source components were found to be chocked due with Rubidium deposition preventing helium beam to come out of exit aperture. The source was opened, cleaned thoroughly and baked and reassembled. Two ampules of 5 grams Rubidium was loaded in Argon atmosphere for charge exchange. Continuous and stable He- beam was achieved with few hours of conditioning. The coolant level was found to be low due to leak. This was fixed with a new set of connectors and chamber externally heated to achieve 400 C. In the month of September, low current from ion source was observed due to chocking. The chocking was cleared externally. After 3 months of operation (nearly 400 hrs) and oven temperature going beyond 2000C decided to go for maintenance and rebuild ion source. The second maintenance in this period was performed in the month of December 2020. After maintenance with 8 hrs of conditioning stable beam was achieved.

Magnet power supply of RF – Charge Exchange Ion Source was fluctuating and due to which the current from source was also not stable. The power supply was replaced by new supply. Since the supply was not of same type, modification had to be done for installing the magnet power supply. The output of supply was stable and the source is now generating stable beam.

#### 1.3.2.2 The 5SDH-2 Pelletron Accelerator and Endstation Maintenance

As the 5SDH-2 was shut down in the lockdown period and it was powered in the month of June. The vacuum in the order  $10^{-7}$  torr was achieved after 2 days of pumping. To know the status of the accelerator, conditioning was done and following observations were made.

1. Terminal voltage (TV) 1.112 MV at CPS 8KV with probe completely out.
2. Tank spark observed at TV above 1.245 MV at CPS 9KV with probe completely out.
3. Stable 1.149 TV observed at CPS 11KV with probe completely out.
4. The probe current 10-20  $\mu$ A at probe position zero.
5. Maximum TV attained is 1.125 MV.
6. No Charge loss/difference in up to down charge was observed.

There were minor issues in the experimental end station. The goniometer operation was not very smooth. It was jumping to some random value during channeling measurements. This problem pops up intermittently. Besides this, end station and Accelerator operated smoothly throughout the year.

## 1.4 AMS AND GEOCHRONOLOGY FACILITIES

### 1.4.1 Accelerator Mass Spectrometry

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An Accelerator Mass Spectrometry facility for the measurement of  $^{14}\text{C}$ ,  $^{10}\text{Be}$  and  $^{26}\text{Al}$ , based on a dedicated 500kV ion accelerator is in operation since March 2015. The 500 kV tandem ion accelerator is procured from National Electrostatic Corp. (NEC), USA. Carbon sample processing and graphitization are performed in a dedicated comprehensive graphitization laboratory while  $^{10}\text{Be}$  and  $^{26}\text{Al}$  samples are processed in a clean chemistry laboratory. Due to ongoing pandemic time, it is difficult to present users physically. Therefore, we have given users an option to send their samples and we are preparing their samples and performing their measurements.

#### 1.4.1.1 Graphitization Laboratory

Graphitization laboratory is equipped with three Automated Graphitization Equipment (AGE) which are coupled with three elemental analysers for the graphitization of organic samples and one carbonate handling system (CHS) for the graphitization of carbonate samples. The graphitization laboratory is routinely utilized for the sample pre-treatment and graphitization of charcoal, wood, macrofossils, plant remains, sediment, bones, textile and carbonate samples (shells, foraminifera). During April 2020-March 2021, 611 samples have been pre-treated and graphitized by 22 users from different universities and institutes for their research work. An operational manual for this laboratory comprising of sample specific pretreatment protocols and graphitization procedures has also been prepared during lockdown period. Beside the routine maintenance of refilling the different tubes in the graphitization equipment, one magnetic valve was also replaced in one of the elemental analysers of one of the AGE.

##### 1.4.1.1.1 Optimization of background value for the graphitization of dissolved inorganic carbon (DIC) using carbonate handling system (CHS)

A method to graphitize dissolved inorganic carbon (DIC) in ground water samples have been developed using carbonate handling system (CHS) last year. Secondary standards (DICS and DICB) were prepared by dissolving IAEA C2 and C1 samples in acidic water, respectively. Measurement results for dissolved C2 samples were matching with its nominal value but dissolved C1 sample was showing higher background value (pMC = 1.09). After studying the methods from other radiocarbon dating lab, it was observed that residual carbon in deionized water (MQ water) may be one of the reason for the higher blank value. To remove this residual carbon, acidified water was heated for 24 hrs at 90°C temperature and then C1 sample was dissolved into it and graphitized using CHS. After addition of this step in the method, background value was reduced to 0.62 pMC.

##### 1.4.1.1.2 Graphitization of dissolved organic carbon using carbonate handling system

A method is being developed to graphitize dissolved organic carbon using carbonate handling system (CHS). For initial experiment, Standard (DOC\_OXII) was prepared by dissolving OXII (5.5mg) in 21 mL MQ water in three 12 mL vials. We have utilized wet oxidation technique to oxidise the dissolved organic carbon in the sample. 1 ml aliquot of acidified sodium persulfate solution (50 mL  $\text{H}_2\text{O}$  + 2g  $\text{Na}_2\text{S}_2\text{O}_8$  + 400 $\mu\text{L}$  of 85%  $\text{H}_3\text{PO}_4$ ) was added as an oxidant to this sample. Sample was flushed with helium gas then heated at 90°C temperature and evolved  $\text{CO}_2$  (329 micro gram) was graphitized using AGE. Measurement result of this sample was close to the results of combusted OXII samples. We will assess the background value of this method and test some real secondary standards with some real samples in the next step.

##### 1.4.1.1.3 Graphitization of methane from atmospheric air samples

A method is being developed to graphitize methane from the atmospheric air samples. For initial experiment, two samples  $\text{CH}_4$ \_Cyl and  $\text{CH}_4$ \_Air have been used. Sample  $\text{CH}_4$ \_Cyl is taken from a cylinder of mixture gas (10%  $\text{CH}_4$  and 90% argon) and sample  $\text{CH}_4$ \_Air is taken from compressed air. These samples were passed through a glass tube containing sicapent, sodalime and zeolite. Sicapent is used to absorb moisture, sodalime is used to absorb  $\text{CO}_2$  and zeolite is used to absorb the traces of  $\text{CO}_2$  from the samples. Remaining gas from the sample is heated at 920 °C over the surface of copper oxide. This heating converts the methane into  $\text{CO}_2$  and water vapours are produced as the by-product in the reaction. Water vapours were again absorbed on sicapent

and CO<sub>2</sub> is absorbed on the zeolite. This zeolite is heated at 550°C and released CO<sub>2</sub> is graphitized using AGE. Sufficient CO<sub>2</sub> (905 micro gram) is obtained after half an hour sampling of CH<sub>4</sub>\_Cyl sample but sufficient amount CO<sub>2</sub> could not be produced from the CH<sub>4</sub>\_Air for same time of sampling. Sample CH<sub>4</sub>\_Cyl is measured successfully in the XCAMS. We will assess the background of this method and also measure some other samples for testing in the next step.

#### 1.4.1.2 XCAMS facility

##### 1.4.1.2.1 AMS Measurements

The compact <sup>14</sup>C Accelerator Mass Spectrometer eXtended for <sup>10</sup>Be and <sup>26</sup>Al (XCAMS) is routinely utilized for the measurement of <sup>14</sup>C, <sup>10</sup>Be and <sup>26</sup>Al. Total 777 samples of <sup>14</sup>C and <sup>10</sup>Be have been measured this year. 32 users from different institutes have utilized this facility for their research work.

##### 1.4.1.2.2 <sup>14</sup>CAMS measurements of samples from industries

IUAC has opened its door to industries for radiocarbon AMS measurements from this year onwards. Initially these measurements are allowed without charge basis for three to four samples. The modus operandi of handling these samples will be worked out by IUAC group regarding the feasibility of measurements. Results will be provided in either print form or email to the industries but no certificate will be provided till IUAC receives the accreditation. Radiocarbon measurements of six samples from two industries have been done this year.

##### 1.4.1.2.3 Maintenance activities of XCAMS

Following maintenance activities were carried out in this facility.

- i. 40 MC SNICS was opened for breakdown maintenance in March, August and January. Cathode voltage was following the immersion voltage. Source was cleaned, re-assembled and brought back to operation
- ii. 134 MC SNICS was also opened for breakdown maintenance. Extractor voltage was fluctuating after reaching at 15 kV. Ionizer current was also fluctuating.
- iii. It was observed that protection cover of ionizer was touching the Einzel lens assembly. This issue was fixed but current was not observed at FC even after maximum ion source parameters.
- iv. Fresh Cs was loaded. Einzel lens assembly was aligned with the help of alignment kit and fixed in the line and source was brought back to operation.
- v. Input voltage module (D/A converter) in process control station of 134 MC SNICS was replaced with new one.

#### 1.4.2 National geochronology facility

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**The objective of the project is to develop a comprehensive Geochronology facility at IUAC that will permit measurement of quality isotopic data for Geochronological purposes including relevant characterization at the highest international level.** The dedicated geochronology facility at IUAC will enable researchers from Indian Universities and research institutes to study different aspects related to Earth Sciences. The proposed geochronology facility will enhance the research capabilities in the country with the following objectives:

- ❖ Carrying out various research studies in the field of climate change, palaeo-climate studies, global carbon cycle, oceanographic parameters, Antarctica research programs, archaeology, biomedicine and history of art etc.
- ❖ Capacity building: Initiation of new PhD programs using the facility for universities and research institutions.
- ❖ Generating geochronological data that shall be of interest to Earth Scientists, which require precise geochronology.

Following instrumentation have been commissioned and being utilized under this project:

#### 1.4.2.1 Quadrupole- Inductively coupled plasma mass spectrometry (Q-ICPMS)

The Q-ICPMS instrument is a very efficient and convenient tool for elemental analysis at ppb level. The Q-ICPMS system equipped with auto-sampler was installed in 2018 and is being used regularly since then. During March 2020 – February 2021, this facility was used for analysis of about 550 samples for 09 users from different institutions. The major thrust areas of research included characterization of spinifex-textured komatiite from Bundelkhand craton (Allahabad University), sediments characterization to study rock weathering (IIT Roorkee), levels of heavy metals in ground water (CSRD, JNU), climate role on soil formation (Aligarh Muslim University), role of micronutrients in pre-term births (THSTI, Faridabad), depiction of provenance and weathering using loess-paleosol samples (IUAC, Delhi), distribution of trace elements and nutrient profile testing in bio-char samples (Central University of Punjab), etc. In addition, the facility was also used for precise estimation of Be and Al in quartzite samples as partial requirement for AMS measurements for surface exposure studies.

Along with regular measurements, attempts were carried out to improve sensitivity of the instrument for trace or ultra-trace level elements (1-10 ppb). In addition, kinetic energy discrimination (KED) mode was utilized to remove spectral interferences generated from plasma gas i.e. Ar. The use of KED mode greatly improved the measurements for elements like Se, Te, Pb, etc. Also, to remove elemental deposition in introduction assembly and cones, the spares were cleaned and re-assembled. The system was tuned using standard solution and mass calibration was done to remove any mass drift observed after re-assembling of spares.



Fig 1: Quadrupole Inductively Coupled Plasma Mass Spectrometer

#### 1.4.2.2 Femto-second laser ablated High Resolution Inductively coupled plasma mass spectrometer

High resolution single collector ICP-MS system at IUAC is coupled with femtosecond Laser Ablation (model: CETAC-Teledyne) and is in use for isotopic ratio measurements, especially for U-Pb dating of minerals. The instrument can also be used in solution mode and connected with an autosampler having 10 standard samples and 120 samples slot capacity for doing sequence of samples in liquid mode automatically. System is also coupled with Prefast auto-dilution system for upto 400x precise dilution. System was optimized for laser spot by ablation of different Zircon grains at varied laser energy. Many measurements in laser ablation mode were done using standards such as Plesovice, 91500 and Temora. The results were in good agreement with high accuracy and repeatability. Zircon samples by IIT Bombay group collected from the Brahmaputra valley were analysed for provenance studies. Later, post lockdown the instrument faced some problems, which were identified and being rectified.

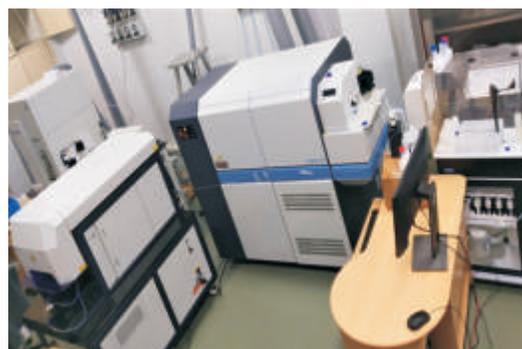


Fig 2: HR-ICPMS coupled with Fs Laser and Prefast auto-dilution system.

#### 1.4.2.3 Field Emission Scanning Electron Microscope (FE-SEM)

The state-of-the-art Field Emission-Scanning Electron Microscope (FE-SEM) is equipped with multiple detectors including Energy Dispersive X-ray spectrometer (EDX), Electron Backscatter Diffraction (EBSD) and Cathodoluminescence (CL) attachments and enables advanced analysis of various sample types. FE-SEM can operate at voltages ranging from 0.1 kV to 30 kV and at magnification ranging from 25× to 1,000,000×, enabling high-quality imaging and analysis of micro and nanostructures.

Non-conductive geological (soils, quartz...etc.) and materials samples (ceramic, polymers...etc.) require carbon and/or metal coating to enable the imaging of samples. Electrically conductive coating (nano thin layer of conductive metal) allows imaging at higher energies with the highest resolution and magnification without concern of sample charging.

Geological, Palaeontological, Nuclear targets and Material science samples are studied by using FE-SEM. 15 users with 239 samples from all over India have been entertained. FE-SEM images of separated clay fractions taken by JEOL JSM 7610F in IUAC is shown in figure 1. These images along with EDX helped the user in the identification of the different clay minerals supporting XRD data.

### Maintenance

The FE-SEM facility worked all-round the year with multiple issues. Following issues have been detected during the operation of FE-SEM.

- LAN port of FE-SEM was found faulty and it was connected directly to HUB. After this, FE-SEM imaging worked perfectly fine.
- Vacuum leak was found in the EBSD detector and issue is still pending.
- LAN port of EDS and EBSD was found damaged. There is a communication problem between EDS and EBSD with FE-SEM.
- FE-SEM chiller was repaired and coolant gas was filled into the compressor.

Users from Delhi University, Amity University, Noida, TIET, Patiala, IUAC, New Delhi, Pondicherry University, JNU, New Delhi, Dayalbagh Educational Institute, Agra, CSIR-NPL, New Delhi, and DCRUST, Murthal used the facility.

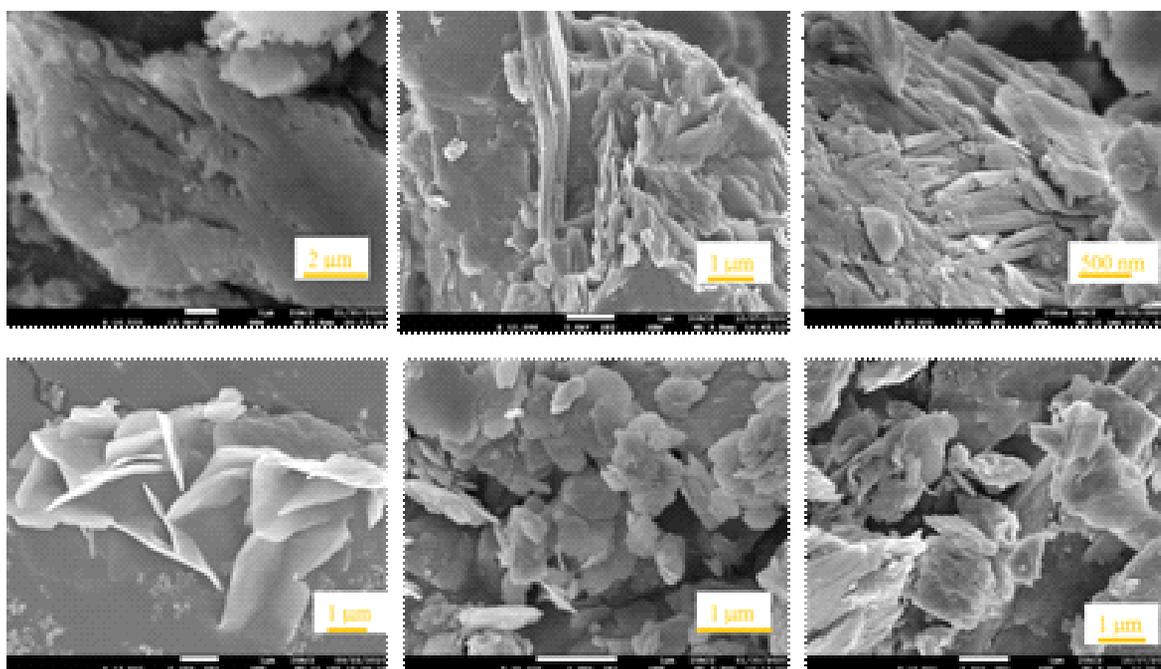


Fig 3: Morphological studies of separated clay fractions.

#### 1.4.2.4 Wave length Dispersive X-Ray fluorescence

A wavelength dispersive X-ray fluorescence spectrometer (PANalytical AXiosmAX) has been installed and operated at IUAC since 2018. Sample preparation for measurements is done using a pellet press, fused bead machine and vibratory cup mill. This non-destructive technique is used to analyse the chemical composition of different geological samples. These quantitative studies help users in geochemical analysis and to study different surface earth processes, the behaviour of contamination in soil and to study provenances. The technique is also used for atomic physics studies to study the chemical shifts in Thallium metal compounds and to relate these shifts to various fundamental parameters. The shift in the transition energy for  $L\gamma_4$  and  $L\gamma_5$  lines of various Thallium compounds with respect to the pure Thallium target is shown in figure 4.

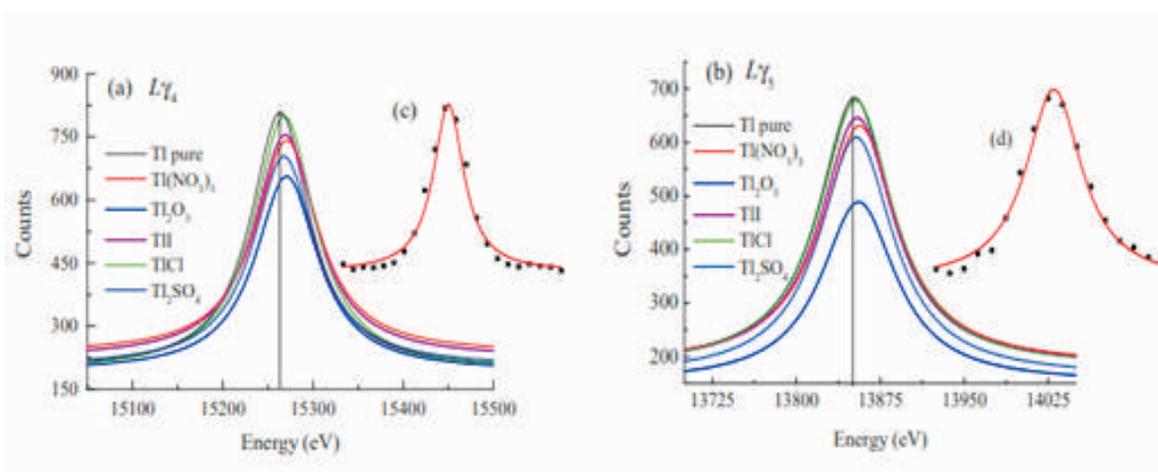


Fig. 4: Chemical effect on  $L\gamma_4$  and  $L\gamma_5$  X-ray lines in Thallium complexes.

From April 2020 to March 2021, the facility has been used for the measurements of 82 samples of 6 users from 3 different universities and 2 institutions.

### Maintenance

The rate of the gas flow of gas proportional counter was not stable and was beyond the allowed limit. The problem was solved with replacement of the electronic controller of the flow detector.

### 1.4.2.5 X-Ray Diffraction

An X-ray diffractometer (PANalytical Empyrean) using non-destructive powder diffraction method has been installed and operated since 2017 at IUAC. The facility was in regular operation round the year except during the lockdown period. From April 2020 to March 2021, the facility has been used for the measurements of 595 samples from 20 users from 7 different universities and 4 institutions.

The facility is used to study the lattice structure or mineralogy of different geological samples (e.g. rock powders, soil, sediment, separated clay fractions, etc.) collected from different locations or for phase identification in the synthesized crystalline materials. The data obtained helped users to calculate the different parameters such as grain size, crystallinity, strain, roughness, etc. for the mineral/phase present in the sample. These analyses helped to understand the phase formation and transformations of the geological samples under different Paleoclimatic changes, depositional environments and earth surface processes, etc.

For XRD and XRF users from Central University of Punjab, IUAC, New Delhi, Amity University, Noida, Delhi University, Panjab University, BHU, Varanasi, BSIP, Lucknow, Jamia Millia Islamia, New Delhi, TIET, Patiala, AMU Aligarh, JNU, New Delhi, NIO, Goa and Punjabi University, Patiala utilized the facility.

### 1.4.2.6 Laboratory magnetic barrier separator

Magnetic barrier separator is widely used instruments for mineral separation on the basis of their magnetic properties. It uses magnetic force and gravitational force (as the path is inclined towards ground) to separate particular mineral from the mixture. The magnetic separator has a large electromagnet through which mineral mixtures is passed on an inclined metal plate. By varying the magnetic field value and/or the slope of the metal plate the minerals of different magnetic properties can be separated from one another. This system has been used by various researchers interested in CRN dating.



Fig 5: Laboratory magnetic barrier separator.

#### 1.4.2.7 Jaw Crusher, Vibratory disc mill and Sieve shaker

These instruments are used for physical processing of rock to convert them in lower grain sizes of powder. More than 150 rock samples have been processed for the purpose of CRN, ICPMS and XRF analysis.

##### Jaw Crusher

Rocks must to broken into suitable sizes prior to crushing. Jaw Crusher is used for crushing large size samples (< 9 cm) to smaller sizes (~ 2 mm). Rock size bigger than 9 cm are broken into small pieces using hammer or any other suitable instrument.

##### Vibratory Disc Mill

Vibratory Disc mill further reduces sample size by grinding it between two cylindrical discs mounted on a circular and horizontal vibratory platform. The feed size of this unit is < 15 mm, which results in output of fine powder of sizes < 20  $\mu\text{m}$ .

##### Sieve shaker

Sieve shaker contains stack of sieves of different pore sizes. The Sieve stack is mounted on the vibratory platform, where the speed and duration of vibration can be adjusted. Sieves of various mesh sizes are used for sieving purposes.



Fig 6: Jaw crusher, vibratory disc mill and sieve shaker.

## 1.5 LOW ENERGY ION BEAM FACILITY (LEIBF)

The COVID-19 pandemic influenced the functioning of every sector worldwide and IUAC was not the exception. Being inter university centre of University Grant Commission (UGC), IUAC also had tough time to meet the requirements of users especially when it along with all universities had to face the standard operating procedure (SOP) guidelines of ministry of home affairs (MOA) during lockdown. The performance of 10 GHz Electron Cyclotron Resonance Ion Source (ECRIS) based Low Energy Ion Beam Facility (LEIBF) [1-3] in the time of crisis has been satisfactory. The breakup of time sharing in major lab activities is shown below in figure 1.

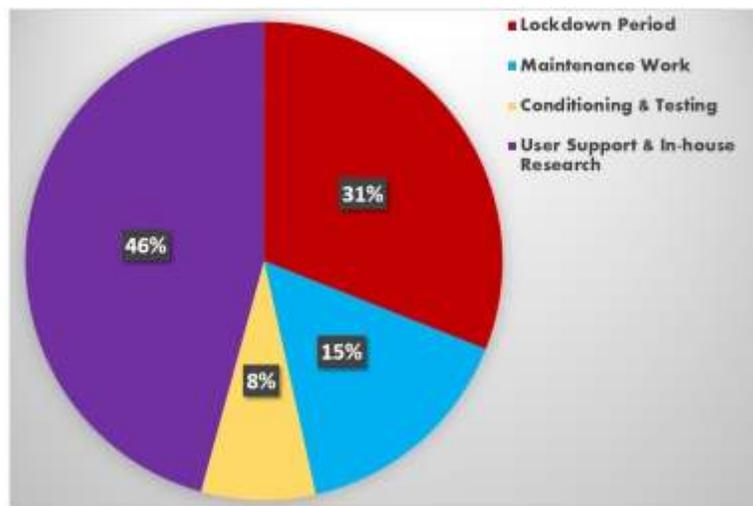


Fig 1: Annual performance of LEIBF at a glance.

About 46% time of present academic year was available for user support. Due to travel constraints, the local users, who were ready with sample preparations, were given opportunities to perform their projected research work and around 80 shifts of beam time was utilized for this purpose. As continued in-house research programs, we conducted a few ion-matter interaction studies on ZnO and Cu-ZnO composites thin films and demonstrated that the transport and optical properties of these films can be tuned by optimizing beam parameters such as ion fluence, beam energy, etc. The experiments with pure and mixed Ne and Xe ECR plasma to address hitherto unrevealed physical phenomenon of gas mixing and isotope anomaly were also conducted. To the best of our knowledge, we could get first data on the abundance dependent inversion of isotope anomaly in pure and  $N_2$  mixed Xe ECR plasma. In natural gas, six stable isotopes of Xe viz.  $Xe^{129}$  (26.44%),  $Xe^{130}$  (4.08%),  $Xe^{131}$  (21.18%),  $Xe^{132}$  (26.89%),  $Xe^{134}$  (10.44%) and  $Xe^{136}$  (8.87%) have different abundances. Recent results of pure and mixed (with  $O_2$  and He) Ne ECR plasma [4] on account of isotope anomaly motivated us to re-perform studies with Xe ECR plasma, but with all six isotopes. The previous studies with three highly abundant isotopes viz.  $Xe^{129}$  (26.44%),  $Xe^{131}$  (21.18%) and  $Xe^{132}$  (26.89%) showed no signature of the isotope anomaly [5]. Theoretically, as fraction of heavier isotope increases, the isotope anomaly in ECR plasma disappears. However, the present experiment demonstrates that if the fraction of heavier isotope is large enough, it can invert the trends of anomaly as depicted in figure 2.

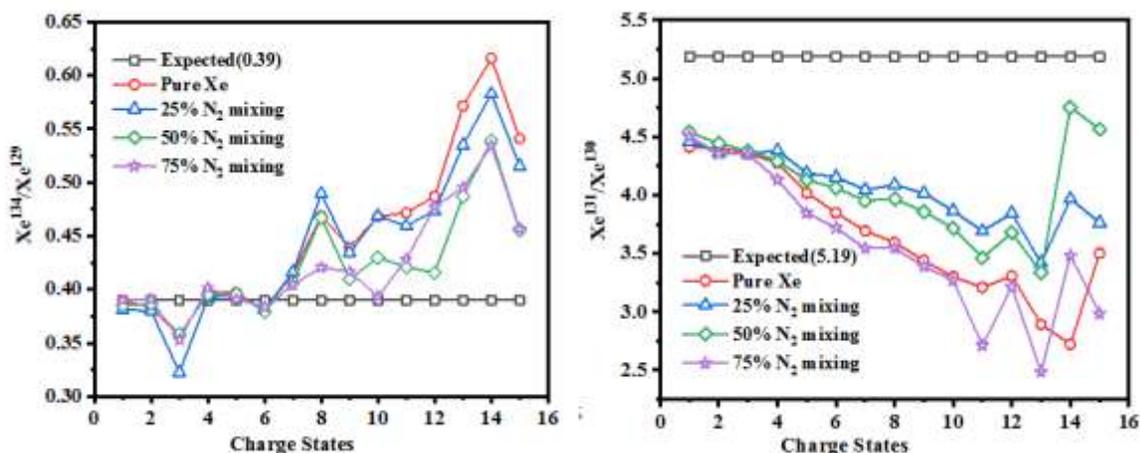


Fig 2: Abundance dependent inversion of trends in isotope anomaly in Xe ECR plasma.

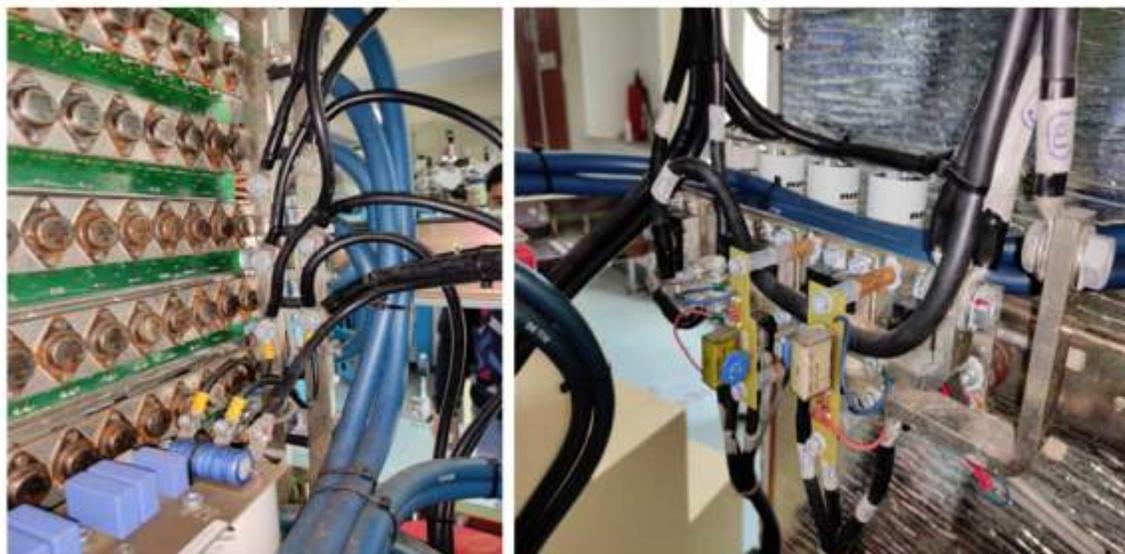


Fig 3: Rusting on the electronic components and chassis of the magnet power supply and magnetic steer/scanner installed in the beam line.

After shutdown of LEIBF during lockdown, the efforts were put to make it live for regular user experiments. The malfunctioning of the magnetic components and high voltage power supplies were seen two major issues with the extraction of beam and its analysis using dipole magnet. The circulation of chilled water in the body channels of magnetic components when they were at power OFF caused rusting on the chassis of the modules and also on the metal holders of electronic components as shown in figure 3. Appropriate maintenance steps were employed by beam transport group to rectify this problem. The high voltage on deck was not stable (a few kV of variation) and it resulted in the difficulty in beam tuning during tests and conditioning. The high voltage stacks were cleaned with de-ionized water and then series of performance tests were conducted by the support team (as depicted in figure 4). The 10 GHz @ 200 W TWT RF amplifiers were also tested with dummy load to ensure minimum reflected power at its high power operation.



In order to generate human resources for sustainability of ECR ion source related futuristic research programs in the country, a two days school on “ECR Ion Source Technology: Opportunities and Challenges” was conducted during 5-6 November, 2020 at IUAC in online mode. The national and international experts shed light on latest development of on the ECR ion source technology and its utilization in nuclear energy production, understanding unknown astrophysical processes and hadron therapy for the treatment of cancer.

We sincerely thank the staff of all support labs and associates of LEIBF for taking challenges in new normal conditions and contributing in their best capacity. All of us have perhaps learnt the importance of co-existence.

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## 1.6 LOW ENERGY NEGATIVE ION IMPLANTER FACILITY

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### 1.6.1 Operation

The pandemic disturbed the operation and experimental activities of low energy negative ion implanter facility. In the beginning of the academic year, from March to May '2020, the facility had to be completely shut down. Thereafter, on 26<sup>th</sup> May, 2020, the whole beam line was powered and the pumps were turned on to create vacuum. The whole beam line, two high voltage decks, high voltage multiplier stack were cleaned. Each beam line component in the facility was checked for their normal operations. Initially, the operation mainly focused on the conditioning of the machine. The ion source had cathode contact problem because of its floating type of connection and general purpose accelerating tubes were not holding high voltages. Once the cathode voltage achieved stability in operation and GP tubes could hold a high voltage of 100KV without any fluctuation, the operation of accelerator started. The operation was mainly focused on laboratory development activities i.e. development of new ion beam species which were of user's experimental demand and improvement in ion beam intensities of known ion beams. User's entry at IUAC facility was restricted during that period. So, this work continued for almost three months, June, July and till mid-August. However, IUAC initiated a program of receiving user's samples through post and conducting the ion beam experiments by IUAC academic staff, and the ion implanter accelerator facility was operational for implantation experiments from **20<sup>th</sup> August, 2020 onwards**. Proper planning, prior communications were made with the respective users. The samples were implanted/irradiated as per their demand i.e. flux rate, fluence and proposed ion depth profiles. Accordingly, the calculations of time and energies for the implantations were done. Handling of samples i.e. loading and taking them out after implantation was done with utmost care. Implanted samples were packed and sent to the respective users by post. In some cases, users collected them from the IUAC gate. Altogether, there were 7 runs of 6 users from different universities. The particulars about the implantation experiments performed during this period are given below:

- a) No. of users: 6
- b) Number of beam run: 7
- c) Total number of shifts utilized/beam on the target: 10
- d) Total number of samples implanted: 150
- e) Ion fluencies used:  $1 \times 10^{13}$  to  $1 \times 10^{16}$  ions/cm<sup>2</sup>
- f) Ion species utilized: <sup>63</sup>Cu, <sup>107</sup>Ag, <sup>197</sup>Au
- g) Energy range utilized : 50 to 100 KeV

### 1.6.2 Maintenance

Preventive maintenance and breakdown maintenance were performed in the academic year 2020-2021. The facility experienced numerous breakdowns in this academic year such as failures of control system, ion source related troubles, GP tubes unable to hold high voltages and magnet power supply tripping and its polarity changing. All of them were rectified.

#### Preventive Maintenances

Preventive measures are essential in regular interval to avoid any unwanted breakdown due to dust and dirt. The outer surfaces of ion source, einzel lens, accelerating tubes and high voltage deck were cleaned from time to time. Thorough cleaning of HV Cockcroft Walton Voltage multiplier stage and accelerating tubes using alcohol were done; thereafter, they were dried with compressed air. It led to improvement in high voltage holding capability of general purpose accelerating tubes to a maximum voltage of 180 KV with stability. The magnet power supply coolant lines were cleaned to have effective cooling. Since the existing GP tubes of negative ion implanter beam line got stains and showed poor performance, it was decided to regenerate the dirty spare GP tubes of old ion source of Pelletron accelerator system. Thereby, 4 numbers of GP tubes were sandblasted, kept immersed in alcohol overnight, dried and baked at 100 °C for one hour and tested. The ceramic gaps of the GP tubes showed improvement up to few hundred Giga ohms range from tens of Mega Ohm range.



Fig 1: From left to right, dirty GP tube, regenerated tube and cleaned Cockcroft Walton Voltage multiplier stage.

### Breakdown Maintenances

The current display of the high voltage power supply controller showed current of very high values that fluctuated randomly from 600 to 800 mA with zero voltage set. The display was found faulty. In the ion source, the cathode current drew high currents to a maximum value of 15mA with lots of fluctuations. The high voltage controller was checked. The connectors from both the cathode power supply end and the load end were removed and cleaned. The cable was checked. Initially, the floating type connection of the cathode with spring caused the cathode spark, but it improved its stability with conditioning. The magnet power supply experienced tripping often and polarity changed during beam run. New cards were made and they replaced the old ones.

During GP tubes cleaning, the coolant pipes of ion source broke and coolant flowed out and spilled all over the surface. Coolant line was laid out with new pipes and new connectors. LOBS coolant filled in the coolant reservoir and coolant system was tested for any leak. The room was cleaned. In vacuum system, MKS vacuum gauge at ion source HV deck was found faulty; it displayed  $\sim 2 \times 10^{-3}$  Torr while other beam line areas, the vacuum were  $\sim 5 \times 10^{-8}$  Torr. For running the ion source, the nearest vacuum gauge to the ion source, MKS gauge of Turbo Pump, TP\_01 output needs to be taken in control console page. Data base was checked. Cables were traced and checked. The necessary connections were made with FGC-011 cable for vacuum reading and vacuum read back was taken to the control console; accordingly, the data base was updated. The Scroll pump used for creating rough vacuum of the target chamber made a lot of sound. The pump is also used as backing pump of Turbo pump which was connected to the chamber. It was replaced with the spare one.

In control system, the read back of ion source parameters showed maximum values while the actual ones at the load side were near zero value. Both ADC and DAC modules were found faulty. Both were replaced with spare ones. In one occasion, the beam line ADC and DAC modules were found off by itself repeatedly; by changing VME crate solved the problem. The client computer stopped working; the control system made functional after reinstalling all the programs to a spare computer.

### 1.6.3 Development Activities

**New Beam Development:** We attempted to develop and improve ion beam current intensities of Te, Sn, Sm, Nb and Pb beams. The cathodes were prepared as per available literature. The updated available ion species from the negative ion implanter facility along with their delivered ion beam intensities are listed in table 1; whereas molecular ion species are listed in table 2.

**Table 1: Available ion species along with their delivered ion beam intensities.**

${}^7\text{Li}$ 100nA	${}^{11}\text{B}$ 400nA	${}^{12}\text{C}$ 1 $\mu\text{A}$	${}^{16}\text{O}$ 1 $\mu\text{A}$	${}^{27}\text{Al}$ 130nA
${}^{28}\text{Si}$ 1 $\mu\text{A}$	${}^{31}\text{P}$ 1 $\mu\text{A}$	${}^{32}\text{S}$ 200nA	${}^{48}\text{Ti}$ 400nA	${}^{56}\text{Fe}$ 150nA
${}^{58}\text{Ni}$ 1 $\mu\text{A}$	${}^{59}\text{Co}$ 1 $\mu\text{A}$	${}^{63}\text{Cu}$ 1 $\mu\text{A}$	${}^{107}\text{Ag}$ 1 $\mu\text{A}$	${}^{197}\text{Au}$ 1.1 $\mu\text{A}$
${}^{74}\text{Ge}$ 500nA	${}^{130}\text{Te}$ 950nA	${}^{93}\text{Nb}$ 23nA		

**Table 2. Molecular ion beams with their delivered ion beam intensities**

$^{24}\text{C}_2$	1.1 $\mu\text{A}$	$^{40}\text{MgO}$	100nA	$^{49}\text{TiH}$	1 $\mu\text{A}$	$^{54}\text{CrH}_2$	1 $\mu\text{A}$	$^{145}\text{SrF}_3$	70nA
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### A trial run of ion implantations at glancing angle

The depth of ion implantation can be reduced by two means; 1) reducing ion energy, and 2) implantation at a glancing angle. We planned to have glancing angle of irradiations to decrease the depth of implantation in the samples. Aluminium wedge with two glancing angles  $70^\circ$  and  $80^\circ$  was fabricated and tested using paper tapes to observe the uniformity of the beam impression. These tapes were exposed to 100KeV,  $^{107}\text{Ag}$  beam for 5 minutes each at an additional incident beam angle at  $2^\circ$ ,  $7^\circ$ ,  $12^\circ$ ,  $17^\circ$  in steps of  $5^\circ$ .  $7^\circ$  is zero angle of incidence in our case). Except for  $2^\circ$ , all impressions in the  $70^\circ$  showed good uniformity. A user had utilized the  $70^\circ$  wedge for 30KeV  $^{16}\text{O}$  implantation in Nickel Oxide sample.