

**THE PNEUMATIC CARRIER FACILITY IN DHRUVA REACTOR : COMMISSIONING,
CHARACTERIZATION AND UTILIZATION**

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60	<i>Abstract :</i>	The 100 MWt power Dhruva Research Reactor, BARC is provided with a Pneumatic Carrier Facility (PCF) to carry out R&D work using short-lived (seconds to minutes) radioisotopes in the fields like Neutron Activation Analysis (NAA) and nuclear fission. The samples are kept inside a high density polypropylene capsule (rabbit), which is pneumatically sent to the irradiation position in the core and retrieved after a preset time of irradiation. After the irradiation, radioactivity assay is carried out using high resolution gamma ray spectrometry with HPGe detector coupled to PC based MCA. The availability of high neutron flux ($\sim 5 \times 10^{13} \text{ cm}^{-2} \cdot \text{s}^{-1}$ at 50 MWt power) and shorter retrieval time (~ 5 seconds) make it possible to measure short-lived isotopes with enhanced sensitivity. This report describes the salient features of this facility, characterization of the neutron spectrum at this irradiation position and its utilization. The PCF is being extensively utilized for analytical applications using NAA as well as nuclear fission studies. A brief description of analysis of some samples of geological, environmental and biological origin, nuclear materials as well as reference materials is included in this report. Protocol and check list for carrying out PCF irradiations and gamma spectrometric assay are also given at the end of the report.
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ध्रुव सयंत्र मे वायु दाब चालित वाहक सुविधा : प्रारम्भ, विवेचना और उपयोग

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भावार्थ: अल्प आयु (सेकंड से मिनट तक) रेडियोसमस्थानिकों में न्युट्रॉन संक्रियण विश्लेषण (NAA) तथा नाभिकीय विखंडन (NF) के क्षेत्र में शोध और विकास कार्य के लिये 100 MW तापीय क्षमता वाले ध्रुव शोध परमाणु सयंत्र में वायु दाब चालित वाहक सुविधा (PCF) की व्यवस्था की गयी है । इस व्यवस्था में उच्च घनत्व वाले पॉलीप्रोपीलीन निर्मित कैप्सुल (रैबिट) में नमूने को रखकर वायु दाब के द्वारा परमाणु सयंत्र के गर्भ में स्थित विकिरण स्थान पर भेजा जाता है तथा नियत समय के बाद पुनः वापस लिया जाता है । विकिरण के उपरान्त रेडियोसक्रियता मापन के लिये उच्च विभेदन क्षमतावाले संगणक चालित HPGe संसुचक का उपयोग गामा किरण वर्णमिति के लिये किया जाता है । 50 MW तापीय क्षमता पर उच्च न्युट्रॉन प्रवाह ($\sim 5 \times 10^{13} \text{ cm}^{-2} \text{ s}^{-1}$) और नमूना वापस लाने की अल्प अवधि (5 sec), अल्पायु समस्थानिकों के मापन की सुग्राह्यता बढ़ाती है । इस दस्तावेज (रिपोर्ट) में PCF की मुख्य विशेषता, न्युट्रॉन स्पेक्ट्रम विवेचना और उपयोगिता का वर्णन है । न्युट्रॉन संक्रियण विश्लेषण तथा नाभिकीय विखंडन द्वारा यह व्यवस्था मुख्य रूप से विश्लेषणात्मक कार्यों में उपयोगी है । इस दस्तावेज में विभिन्न प्रकार के स्रोतों से प्राप्त नमूनों तथा भूगर्भीक, पर्यावरणीय, जैविक, नाभिकीय और संदर्भ पदार्थों के विश्लेषण का संक्षिप्त वर्णन है । इस रिपोर्ट के अंत में आवश्यक निर्देश भी संलग्न है।

ABSTRACT

The 100 MWt power Dhruva Research Reactor, BARC is provided with a Pneumatic Carrier Facility (PCF) to carry out R&D work using short-lived (seconds to minutes) radioisotopes in the fields like Neutron Activation Analysis (NAA) and nuclear fission. The samples are kept inside a high density polypropylene capsule (rabbit), which is pneumatically sent to the irradiation position in the core and retrieved after a preset time of irradiation. After the irradiation, radioactivity assay is carried out using high resolution gamma ray spectrometry with HPGe detector coupled to PC based MCA. The availability of high neutron flux ($\sim 5 \times 10^{13} \text{ cm}^{-2} \cdot \text{s}^{-1}$ at 50 MWt power) and shorter retrieval time (~ 5 seconds) make it possible to measure short-lived isotopes with enhanced sensitivity. This report describes the salient features of this facility, characterization of the neutron spectrum at this irradiation position and its utilization. The PCF is being extensively utilized for analytical applications using NAA as well as nuclear fission studies. A brief description of analysis of some samples of geological, environmental and biological origin, nuclear materials as well as reference materials is included in this report. Protocol and check list for carrying out PCF irradiations and gamma spectrometric assay are also given at the end of the report.

1. INTRODUCTION

The Pneumatic Carrier Facility (PCF) in the 100 MWt power Dhruva Research Reactor enables measurements of the activities of short-lived radioisotopes with half-lives of the order of a few seconds to minutes. This facility has been provided in the reactor for carrying out short irradiations required in a variety of studies like neutron activation analysis (NAA), neutron induced nuclear fission and delayed neutron measurements. Samples of different matrices are placed in a polypropylene capsule (rabbit) of suitable dimension, which is used as a sample carrier to the irradiation position and back. The rabbit is pneumatically sent to the irradiation position from sending/receiving (S/R) station located in the Radiochemistry laboratory in the Dhruva reactor complex. The transit times to send the rabbit to the irradiation position in the reactor and to retrieve the same are about 5 seconds, and the permitted irradiation time, which can be preset, is upto a maximum of 999.9 seconds. Neutron flux at this irradiation position, which has been measured as a function of reactor operating power, is about $5 \times 10^{13} \text{ cm}^{-2}\text{s}^{-1}$ at 50 MWt power.

The studies mentioned above involve irradiating target material placed inside the rabbit (dimensions 25 mm dia x 43 mm) in a neutron flux, measurement of activities formed and using these activities to calculate concentrations of the elements in NAA or fission yields in nuclear fission. The sensitivity of this technique is dependent on the neutron flux available, the neutron absorption cross section of the isotope of the element of interest and the efficiency of the radiation measuring detectors. With the availability of large volume High Purity Germanium (HPGe) detectors and the stable sophisticated electronics, PC based MCA and peak area analysis software, reliable analysis of trace elements has been achieved. Using the higher flux and the possibility of retrieving the samples within 5 seconds, it is possible to analyze the short-lived isotopes as well as those isotopes with low cross section values. This facility can effectively be used to determine the concentrations of a large number of elements with enhanced sensitivities with a less turn around time. Irradiation time plays an important role in NAA for determining concentrations of different elements depending on the half-lives of the activation products. Short irradiation helps in faster build-up of radioactivity of short-lived nuclides compared to long-lived nuclides of matrix elements. By optimizing the irradiation and decay times it is possible to selectively measure both short and medium lived

nuclides (seconds to hours half-lives) in the samples of biological and geological origin as well as other samples.

The PCF is being used in NAA of samples of various areas of R&D and analytical applications in nuclear materials like fuel cladding materials and uranium ores, environmental samples like coal, coal fly ash, soil and air particulate matter, life science samples like various parts of plants as well as seeds, geological samples like meteorites and precious stones, forensic samples and archaeological samples. Radiochemistry Division has participated from users' side in commissioning and characterizing PCF and is the principal user since its commissioning. Availability of high neutron flux in the pneumatic carrier facility (PCF) of the Dhruva reactor is advantageous in enhancing the analytical sensitivities [1]. A few experiments were carried out to quantify the analytical sensitivities. Two IAEA reference materials Lichen (IAEA-336) and freeze-dried animal blood (A-13) were analyzed using the PCF irradiations. For most of the elements, two orders of higher sensitivity were achieved by using PCF irradiation compared to Apsara reactor irradiation. Additionally, the higher sensitivities coupled to the access to measure radioactivity with lower cooling times enabled the determination of the concentrations of a few elements in these reference materials that were not reported by IAEA.

In this report, a brief description of the system, glimpses of commissioning experience, and details of the work on the measurement of neutron flux and enhanced sensitivities, and a few applications are presented. In addition, protocol for carrying out irradiations in PCF and a sample irradiation form along with check-list are given as annexes.

2. DESCRIPTION OF THE FACILITY AND COMMISSIONING EXPERIENCE

2.1 Sending and receiving station

The rabbit sending and receiving (S/R) station is enclosed in an airtight β - γ fume hood box to avoid leakage of air, while shooting and retrieving the rabbit. This arrangement ensures that the release of the activity from the rabbit line to the laboratory is minimum. The Pneumatic Carrier Facility consists of a pneumatic carrier rod (PC Rod) in which the rabbit travels between the S/R station and the irradiation position and back by compressed air at a pressure of about 1.5 kg/cm². The schematic of the PC Rod is given in Fig.1. The rabbit is

sent to the irradiation position in PC Rod through SS pipe, which is about 40 meters in length. After carrying out the irradiation for a preset time, the rabbit is retrieved. During the irradiation the rabbit is continuously cooled by compressed air. Continuous cooling of the PC Rod ensures that it is not affected by the gamma ray heating in the reactor core [2,3].

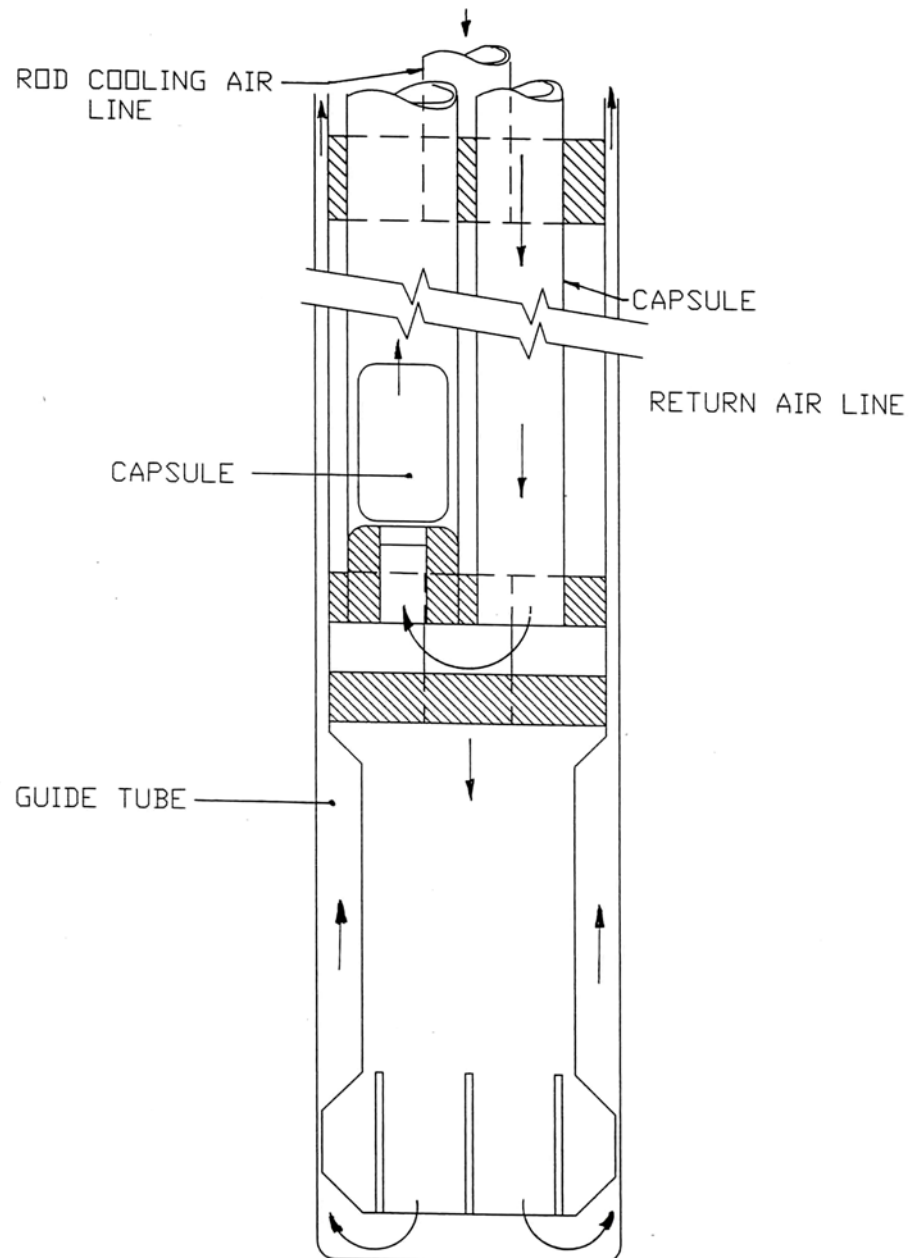


Fig. 1: The schematic of the Pneumatic Carrier Rod

2.2 *Irradiation capsule (Rabbit)*

The material of rabbit is polypropylene of high purity. Rabbit is made by using a pre-designed and qualified mould. The rabbits are of dimensions 25 mm dia x 43 mm with a wall thickness of 2.5 mm. Edges on both the sides were rounded off so as to have a smooth travel in the transit inside the PC Rod. The cross sectional view of the rabbit is given in Fig.2. It has an excellent mechanical strength and its mechanical stability was tested by various drop tests. Differential Scanning Calorimetry (DSC) studies of the rabbit material showed that it gets softened at 165⁰C and this suggests good thermal stability for using this material for the capsule in Dhruva PCF irradiations¹. Repeated and prolonged irradiations of empty rabbit using Apsara irradiations indicated that it has good radiation stability. Small pieces of Rabbit material was analyzed for impurities by NAA using Apsara reactor irradiation and the results showed that impurities like Cl and Al were below detection limits².

2.3 *Control logic of PCF*

The schematic of PCF piping for shooting the polypropylene rabbit, its path from PCF room (Room No.130) to pile and from pile to PCF room after irradiation are shown in Fig. 3. Two optical detectors D1 (located in PCF room) & D2 (located at the reactor upper service space closer to the PC rod) monitor the rabbit position and actuate the solenoid valves. The solenoid valves SV-25 & SV-16 open to send the capsule from PCF room to pile. The capsule, while crossing the detectors D1 & D2, will generate D1 forward (D1F) & D2 forward (D2F) signals respectively. The irradiation timer starts and SV-25 & SV-16 close as soon as D2F is registered. Sample cooling flow starts after a delay by opening SV-22 & SV-29.

At the end of the preset irradiation time, SV-12 & SV-18 are opened and the rabbit is ejected out. Simultaneously SV-22 & SV-29 will close. The return of the irradiated rabbit is done in two steps. The first set of solenoid valves (SV-12 & SV-18) bring the capsule from pile to upper service space and other set of solenoid valves (SV-13 & SV-27) bring the capsule from upper service space to PCF room. This arrangement prevents the leakage of

¹ The temperature at the aluminum seat of the capsule is indicated to be between 50 and 100 ° C, as noted from the change in the colour of the indicating chalk.

² However, later when this material was used as rabbit in PCF of Dhruva reactor, trace levels of Cl and Al were found due to enhanced sensitivities of Dhruva reactor irradiation.

active air from pile to PCF room. The signals D2R & D1R are generated during return of capsule while crossing D2 & D1.

Control logic of the PCF system was designed adopting fail-safe philosophy. System will be brought to safe state in case of anticipated occurrences as mentioned below:

- Failure of solenoid valves: Normally open (NO) type solenoid valves (V-12 & V-18) are used for ejecting out the capsule from pile. In case of failure of solenoid coil the above valves will open and capsule will be ejected out automatically.
- Failure of sample cooling: In case of Lo/Hi flow of sample cooling air the capsule will be ejected out of pile.

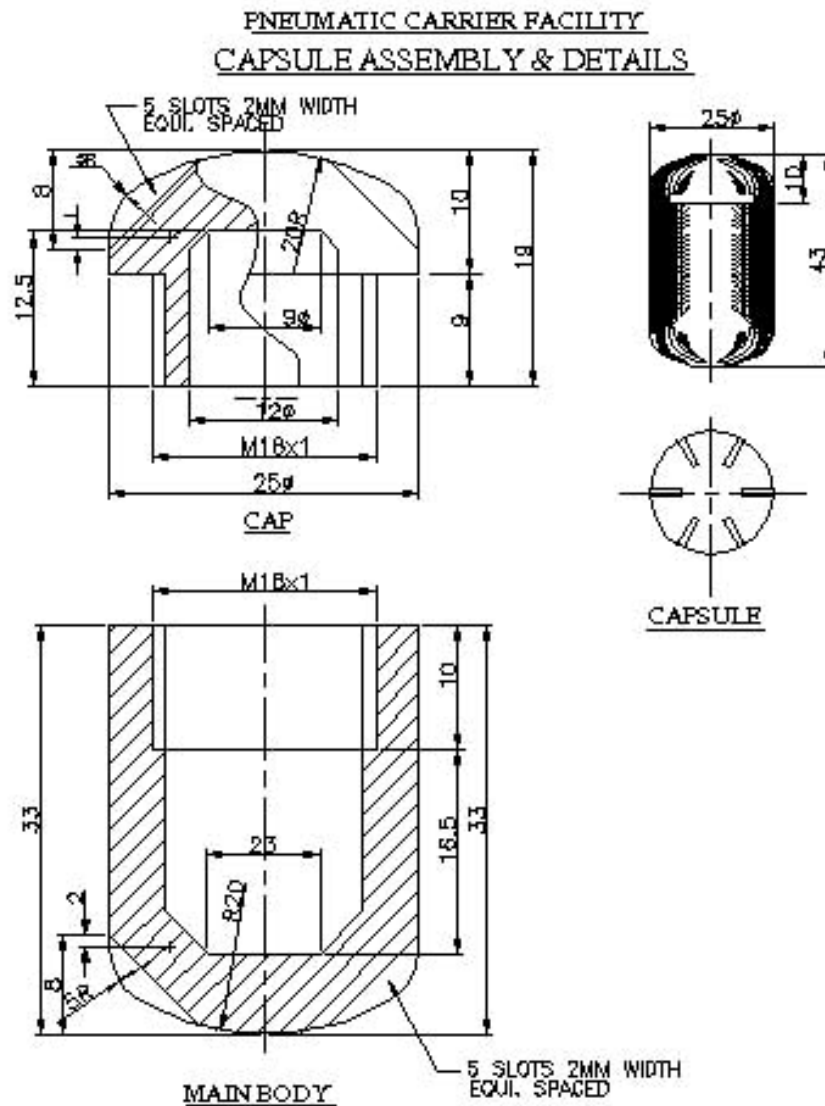


Fig. 2: Cross section view of the Dhruva PCF rabbit

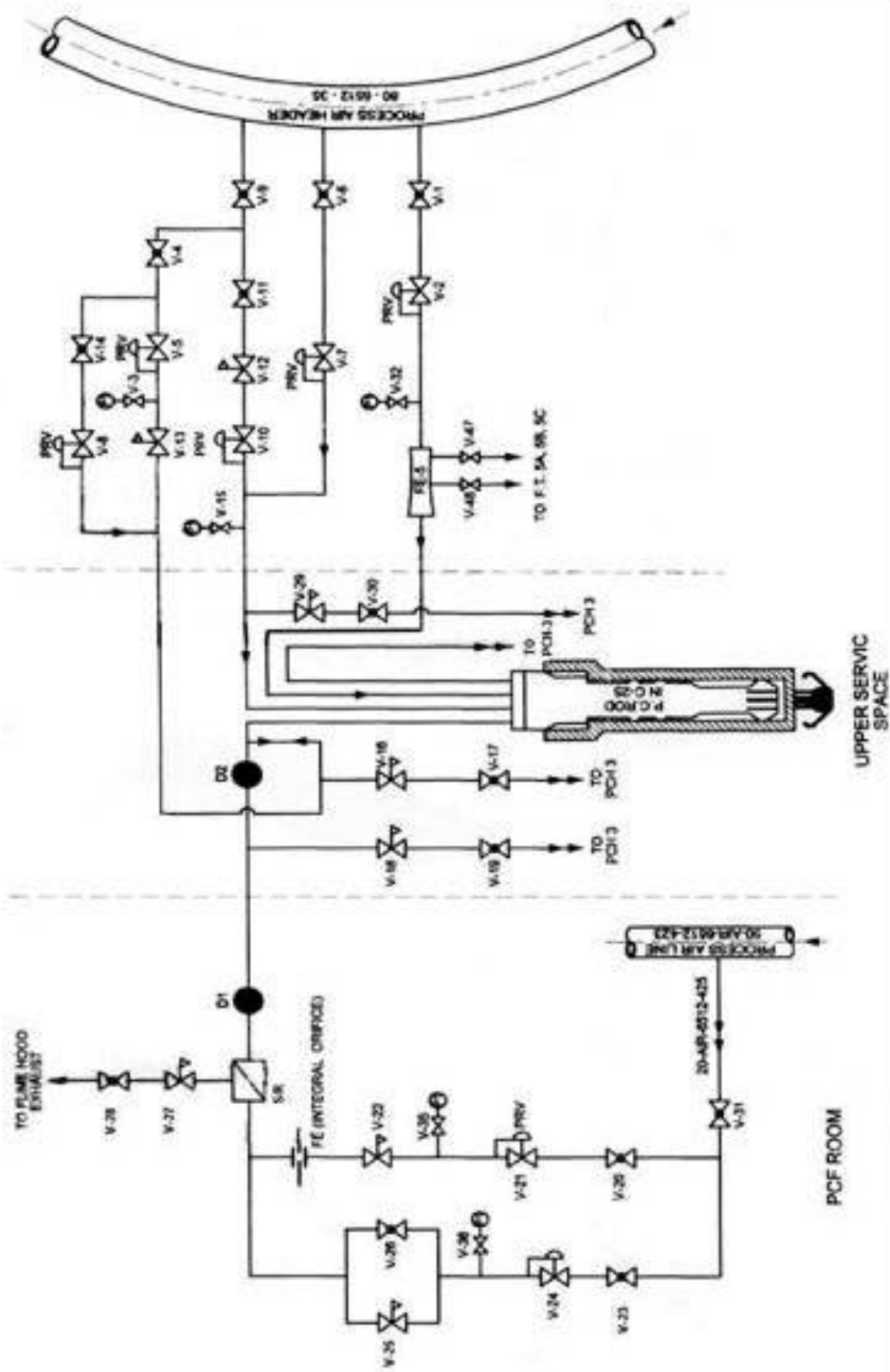


Fig. 3 : Schematic view of the transfer route of rabbit

- Failure of electrical power supply: In case of power supply failure, V-12 & V-18 will de-energize, ejecting out the capsule under irradiation.
- Failure of compressed air: In case compressed air pressure falls below a preset value, V-12 & V-18 will de-energize, ejecting out the capsule under irradiation.

Moreover, provision for manual intervention exists for ejecting sample under irradiation. In case of failure of normal sample ejection line, alternate path through V-7 in parallel with V-12 can be used manually to retrieve the sample. Manual operated valves in parallel to the solenoid valves are also provided for sending the capsule to pile and retrieving to PCF room after irradiation.

2.4 *Detectors and associated circuits*

Optical detectors were used for capsule detection. An electronic signal processing circuit was designed using TTL logic that will detect the light interruption, due to passage of capsule, and generates relay contact change over (forward or reverse) from the sequence. This circuit was populated and tested extensively at different capsule velocities, in shop, prior to installation. The salient feature of the circuit is that, though single detector is used to detect both forward and return movements of the capsule, it distinguishes the direction from the sequence. Up-gradation of the detection system was done by using IR detectors in place of optical detectors. Circuit modification during development stage and subsequent trials helped in optimization between spurious detection and maximum speed of detection.

2.5 *Radiation monitors*

Radiation monitors were installed for personnel protection in pile gallery, in the attached laboratory corridor in the first floor, where the capsule-carrying pipe passes through. Two monitors were installed in the PCF room. The alarms for these detectors were set as per recommendations of safety committee.

2.6 *Modifications based on operating experience*

Based on the initial trials, it was concluded that repeatability of the capsule irradiation at same elevation in pile could not be assured with a continuous capsule cooling flow (40-60 Lpm) before and during the period of irradiation in reverse direction (i.e. from bottom to top) as envisaged in the original design [2]. Subsequently, the mode of capsule cooling air flow was changed from 'reverse' (i.e. bottom to top) to 'forward' (i.e. top to bottom) and the flow was set at ~200 Lpm. Prior to this, various transient analyses were carried out to ensure safety of capsule integrity in pile. Practice of keeping sample cooling ON throughout the period of irradiation was also adopted. Parameters like rabbit shooting pressure, rabbit cooling flow and the direction of cooling flow were optimized, which are being used in further experiments.

3. EXPERIMENTAL

3.1 *Sample preparation*

Known quantity of a gold foil was dissolved in aquaregia and diluted to a definite volume with dilute HNO₃. Aliquots of solutions weighing about 10-20 mg contain about 1-2 µg of gold [4]. Gold standard was prepared by transferring accurately weighed quantities of solution on cellophane paper, dried under IR lamp and sealed in polythene. The Au samples were placed in the rabbits for irradiation for the experiments on neutron flux measurements. Similarly other elemental standards of Al, F, Se, V etc. were prepared.

For measurement of sensitivities, about 20 to 50 mg masses of the samples of CRM were sealed in polypropylene tubes along with a known amount of gold standard sealed separately in polythene and placed in the polypropylene capsules (rabbit). For different studies, representative samples for irradiation were prepared in a similar way and sealed in polythene cover. For each irradiation to monitor neutron flux, a sample of gold target was made, sealed in polythene separately and placed in the rabbit along with experimental samples.

3.2 *Sample irradiation*

For each irradiation, sealed samples and standard were placed in a rabbit and given a unique identification number by operating crew. This rabbit was sent to irradiation position from S/R station. After completion of the irradiation for a preset time of 30 s or 60 s, the rabbit was retrieved from the reactor to the S/R station.

3.3 *Safety aspect*

Expected dose rate, heat generation in addition to suitability of irradiating the material etc. are routinely assessed before irradiation by reactor physicist based on the information provided by the user in the Pile Irradiation Request (PIR) form. Dose rate for each irradiated sample was measured and recorded by the Health Physicist. Depending on the level of contact dose rate, either the capsule was accessed to open or it was placed in a lead castle kept below the S/R station. Both contact dose as well as dose out side the lead container was recorded. It is to be noted that the initial dose from the sample is high due to the presence of short-lived nuclides like ^{28}Al and ^{52}V as well as nuclides of half-lives below 1 min and it decreases rapidly with time. After ensuring that the radiation dose out side the lead container was within the permissible levels the samples were taken out and examined for seal integrity. Integrity of all the irradiated samples was found to be good.

3.4 *Sample preparation for radioactive assay / counting*

Samples were taken out of the outer polythene sealing and then the surface was cleaned with wet tissue paper. Each sample was mounted on a Perspex plate and given a sample identification code. Similarly gold and other elemental standards were mounted and numbered.

3.5 *Radioactivity measurement*

These mounted samples were measured for gamma ray activity by placing them in an efficiency calibrated position using High Purity Germanium detector coupled with a multi-

channel analyzer, PHAST developed by Electronics Division, BARC [5]. The detector has a resolution of 1.9 keV at 1332 keV of ^{60}Co . Gamma ray spectra were accumulated in live time mode and dead time was maintained at $< 5\%$ by placing the sample at appropriate distance from the detector. Gamma ray spectrometer was energy and efficiency calibrated using ^{152}Eu standard source, which has prominent gamma rays in the region of 121.8 – 1408 keV.

3.6 Spectral analysis for peak area determination

Gamma ray spectra were analysed for peak areas under characteristic gamma rays using the software PHAST [5]. The peak shape is taken as a Gaussian with a low energy exponential tail. The background is taken as polynomial background. The software has provision for energy calibration and peak shape/FWHM calibration to determine the peak shape parameters. The coefficients of this fitting are stored and used for subsequent fitting. Least square methods are used to fit the data and the area is then derived from fitted shape parameters. The PHAST software gives fitted peak areas along with the error, which includes errors due to fitting and counting statistics.

4. CALCULATIONS

Radioactivity (A_0) of an isotope formed at the end of neutron irradiation for a period of time t is given as:

$$A_0 = N\sigma\phi (1 - e^{-\lambda t}) \quad (1)$$

where, N = the number of the atoms of an isotope of interest in the target, σ = the neutron absorption cross section of that isotope, ϕ = neutron flux, λ = the decay constant of the activation product and the term $1 - e^{-\lambda t}$ is the saturation factor (S). The activity (A) after a cooling period of T is given by [6],

$$A = N\sigma\phi (1 - e^{-\lambda t}) e^{-\lambda T} \quad (2)$$

The term $e^{-\lambda T}$ is the decay factor (D). Radioactivity is related to measured counts per second (cps), the detection efficiency (ϵ) for the chosen detector sample geometry at the gamma ray energy and the gamma ray abundance (γ) as

$$\text{cps} = P_A / LT = (N_A \theta m / M) \cdot (\sigma \phi) \cdot S \cdot D \cdot C \cdot \epsilon \cdot \gamma \quad (3)$$

where P_A is the peak area, LT is the live time of counting, $C = (1 - e^{-\lambda \cdot CL}) / \lambda LT$ is the correction factor for decay during counting, CL is the clock time, N_A is the Avogadro number, θ is the abundance of isotope of interest, m is the mass of the element and M is the atomic mass of the isotope. From the measured peak areas and the relevant nuclear data, neutron flux is calculated using eqn. 3. Concentration calculations are usually carried out by relative or k_0 -based NAA as described below.

4.1 *Relative method*

In this method, a multielement standard is co-irradiated with the sample and the activities from both sample and standard are measured in identical geometry with respect to the detector. This method is simple to arrive at the concentration values. Using the mass of the element in the standard ($m_{x, \text{std}}$) and count rates of the standard ($\text{cps}_{x, \text{std}}$) and sample ($\text{cps}_{x, \text{sample}}$), the mass of the element in the sample ($m_{x, \text{sample}}$) is determined by the following equation.

$$m_{x, \text{sample}} = m_{x, \text{std}} \cdot \frac{\text{cps}_{x, \text{sample}}}{\text{cps}_{x, \text{std}}} \cdot \frac{D_{\text{std}}}{D_{\text{sample}}} \quad (4)$$

The $m_{x, \text{sample}}$ (μg) is converted to concentration (e.g., $\mu\text{g} \cdot \text{g}^{-1}$) by dividing with sample mass (g). Though the relative method is simple and precise, prior knowledge of the elements present in the sample is necessary to prepare multielemental standards or use certified reference materials (CRMs) of similar matrices.

4.2 *Single comparator or k_0 -NAA method*

The k_0 -NAA technique involves simultaneous irradiation of a sample and a neutron flux monitor, such as gold (^{197}Au), and the use of a composite nuclear constant called k_0 [7,8].

This method obviates the preparation of standards for each element and a priori knowledge of the constituents of sample is not necessary. The concentration of an element is calculated using the following equation.

$$C(\mu g \cdot g^{-1}) = \frac{\frac{P_A/LT}{S.D.C.W}}{\left(\frac{P_A/LT}{S.D.C.w}\right)^*} \cdot \frac{1}{k_0} \cdot \frac{f + Q_0(\alpha)^*}{f + Q_0(\alpha)} \cdot \frac{\varepsilon^*}{\varepsilon} \quad (5)$$

where, W and w are masses of sample and single comparator respectively, f is the sub-cadmium to epi-thermal neutron flux ratio (f), α is the neutron flux shape factor, $Q_0(\alpha)$ ($=I_0(\alpha) / \sigma_0$) is the α corrected Q_0 , I_0 is the resonance integral above the Cd cut off energy (0.55 eV for 1 mm thick Cd cover) and σ_0 is the (n, γ) cross section for 2200 m/s neutrons, ε is the absolute efficiency of the detector and k_0 is a factor and is defined as:

$$k_0 = \frac{M^* \theta \sigma_0 \gamma}{M \theta^* \sigma_0^* \gamma^*} \quad (6)$$

The symbol "*" refers to the corresponding parameters of the comparator like ^{197}Au . Instead of using computed k_0 values, recommended k_0 values available in literature [8] are used, which were experimentally determined. Details of k_0 -NAA methodology are available in references 4 and 9.

5. RESULTS AND DISCUSSION

5.1 Dose rate measurements

Laboratory R-130 and its adjacent lab (R-129) are provided with air activity monitors. Over the last few years about 300 PCF irradiations were carried out, in addition to dummy test runs and air activity was not observed. As soon as the sample is received, as a routine health & safety requirement, station Health Physicist measures contact radiation dose rate of the irradiated rabbit with a tele-dose meter. Contact dose on most of the empty irradiated rabbits was around 1 R/h where as on irradiated rabbits with geological samples it was 1-2 R/h. But within 3 min, it comes down to permissible levels. The irradiated samples were analysed using activation principle. From the activation analysis of rabbit samples, Al and Cl were

found to be in the range of 50 mg/kg and 70 mg/kg respectively. This explains the dose rates of irradiated rabbits, which weigh about 12 g. Aluminium and Cl in the rabbit samples could not be measured using Apsara irradiation as the sample amount was in the range of 20 – 50 mg and sensitivities in the Apsara irradiations were about 100 times lower (cf: Section 5.4). However, in most of the cases, after a 3 min cooling, dose rates permitted to open the rabbit and retrieve samples for further analysis.

5.2 Neutron spectrum characterization

Neutron activation analysis (NAA) as well as other studies like neutron induced nuclear fission and nuclear reaction cross section determination, often need the value of neutron flux as input. In addition, characterization of neutron spectrum is a prerequisite in many applications. Neutron flux in the PCF irradiation position was determined by standard activation method using Au targets and neutron spectrum was characterized by determining the two neutron flux parameters namely (i) the epithermal neutron flux shape factor (α) and (ii) subcadmium-to-epithermal neutron flux ratio (f) using bare detector methods [9]. The obtained values of f and α are 150 ± 3 and 0.011 ± 0.004 respectively. The high value of f suggests that most of the neutrons in the PCF irradiation position have energy below cadmium cut off energy (0.55 eV for 1 mm thick cadmium) and can be considered as thermal energy neutrons (>99% thermal neutron). Accordingly, thermal neutron absorption cross-section values can be used in the calculations. Low value of α suggests that the shape of neutron spectrum is nearly ideal. The relevant nuclear data used here as well as in the other experiments that follow, have been taken from literature [10] and are given in Table 1.

5.3 Neutron flux measurement

Neutron flux values were determined using gold activation at 10, 20, 30, 45 and 50 MWt from replicate irradiations (about 10 at each power). At each chosen reactor power, targets containing accurate amounts of Au were irradiated for 60s and retrieved.

Table 1: Relevant Nuclear data used in NAA work

Target Nuclide	Isotopic Abundance θ , %	σ_0 (b)	Q_0	Isotope Formed	Half-life ($t_{1/2}$)	Gamma-ray Energy E_γ , (keV)	Gamma-ray Abundance γ , %
¹⁹ F	100	0.0096	2.2	²⁰ F	11.2s	1633.6	100
²³ Na	100	0.513	0.59	²⁴ Na	15.03 h	1368.5	100
²⁶ Mg	11.0	0.0372	0.64	²⁷ Mg	9.5 m	844	71.4
²⁷ Al	100	0.23	0.71	²⁸ Al	2.24m	1778.9	100
³⁵ S	0.02	0.16	1.12	³⁶ S	5.05 m	3103.8	94.1
³⁷ Cl	24.23	0.423	0.69	³⁸ Cl	37.21m	1642.4	32.5
⁴¹ K	6.73	1.45	0.97	⁴² K	12.40 h	1524.7	6.7
⁴⁵ Sc	100	9.8	0.43	^{46m} Sc	18.7s	142.5	56
⁴⁸ Ca	0.19	1.12	0.45	⁴⁹ Ca	8.718 m	3084.4	92.1
⁵⁰ Ti	5.4	0.17	0.67	⁵¹ Ti	5.76m	320.1	93
⁵¹ V	99.75	4.92	0.55	⁵² V	3.75m	1434.1	100
⁵⁵ Mn	100	13.3	1.05	⁵⁶ Mn	2.59 h	846.6	100
⁶⁵ Cu	30.83	2.48	1.06	^{66m} Cu	5.1 m	1039.2	7.4
⁷¹ Ga	39.9	4.61	6.63	⁷² Ga	14.10 h	834	39.9
⁷⁵ As	100	3.86	13.6	⁷⁶ As	26.24 h	559.1	100
⁷⁶ Se	9.37	22	0.77	^{77m} Se	17.4s	162	52.4
¹⁷⁸ Hf	27.28	53	7.9	^{179m} Hf	18.7 s	214.3	95.2
⁸¹ Br	49.31	2.59	19.3	⁸² Br	35.50 h	776.4	49.4
⁹³ Nb	100	0.863	7.35	^{94m} Nb	6.26 m	871.1	0.50
¹⁰⁷ Ag	51.84	33.1	2.9	¹⁰⁸ Ag	2.37m	633	1.76
¹¹⁵ In	95.7	157	16.8	^{116m} In	54.1m	1097.2	56.2
¹²⁷ I	100	4.04	24.8	¹²⁸ I	25m	443	16.9
¹³⁸ Ba	71.7	0.405	0.88	¹³⁹ Ba	83.06 m	165.9	23.76
¹³⁹ La	99.91	9.43	1.24	¹⁴⁰ La	40.22 h	328.8	99.9
¹⁵¹ Eu	47.81	3222	0.57	^{152m} Eu	9.3 h	842	14.2
¹⁵² Sm	26.7	220	14.4	¹⁵² Sm	46.50 h	103.2	22.5
¹⁶⁴ Dy	28.2	1697	0.25	¹⁶⁵ Dy	139.20 m	361.5	28.18
¹⁷⁶ Lu	2.59	2100	34.8	¹⁷⁷ Lu	6.71 d	208.4	100
²³⁸ U	99.27	2.68	103	²³⁹ U/ ²³⁹ Np	2.27 d	277	99.2
²³² Th	100	7.34	11.4	²³³ Th/ ²³³ Pa	26.97 d	311.8	100
¹⁹⁷ Au	100	98.7	15.7	¹⁹⁸ Au	2.7d	411.8	95.58

$\sigma_0(b) - (n, \gamma)$ neutron absorption cross-section at 2200 m/s; Q_0 - ratio of resonance integral (I_0) to σ_0 ;

Gold was chosen as the target material as it is mono isotopic, its σ_0 value is 99 b and has an intense gamma ray at 411.8 keV and its gamma ray spectrum is simple. Targets were taken out of the irradiated rabbit after a cooling period of 3 min, mounted on separate Perspex plates and the activities were measured using high-resolution gamma ray spectrometry. Fig. 4 shows a typical gamma ray spectrum of irradiated Au target. From the spectrum, peak area under the characteristic γ -line of 411.8 keV was obtained and used in eqn. 3 to calculate neutron flux. Measured data and calculated neutron flux values at 20 MWt reactor power are given Table 2. It may be noted that the neutron flux values are having a range of (1.9-2.2) x

$10^{13} \text{ cm}^{-2}\text{s}^{-1}$ at 20 MWt. These fluctuations depend on the position that the rabbit reaches and the level of the moderator in the reactor.

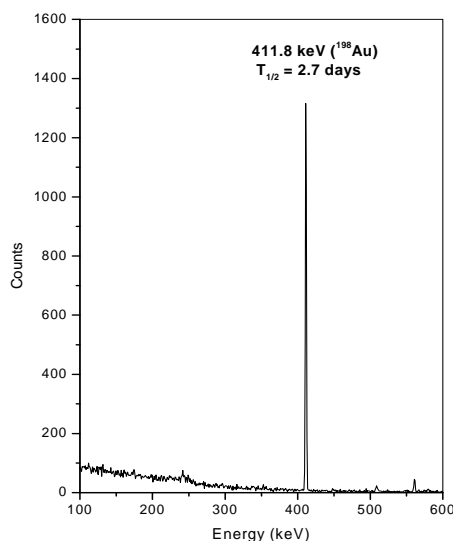


Fig. 4 Gamma ray spectrum of a neutron activated gold (^{197}Au) standard

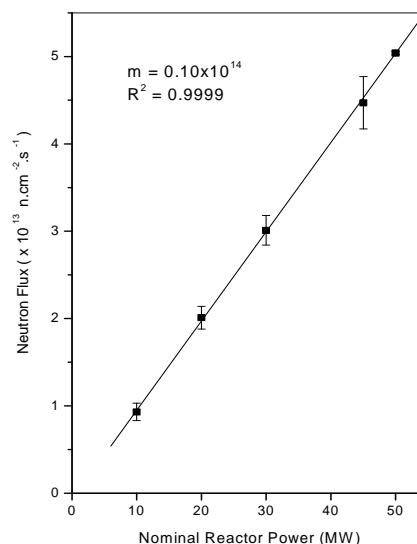


Fig. 5 Measured neutron flux, at the irradiation position of PCF, Dhruva as a function of the reactor power level

To account these fluctuations, it is essential to use a flux monitor like ^{197}Au in each set of PCF irradiations to monitor neutron flux seen by the sample during the irradiation. A plot between the operating power of the reactor (10-50 MWt) versus the neutron flux determined is shown in Fig. 5 demonstrating the linear dependence of flux ($R^2=0.9999$) on the reactor power.

5.4 Evaluation of the methodology: determination of short-lived isotopes

One of the advantages of using PCF irradiation is determination of elements like F, Se, Sc, Hf, Al, V, Ti, Cu, S, Ca and Mg, that have short-lived radioisotopes having half-lives in the range of a few seconds to minutes. To evaluate this possibility and the efficacy of the methodology, standards for Se (element Se) and F (compound LiF) were irradiated, and the activities of Se and F were measured by gamma ray spectrometry. Gamma ray spectra of irradiated samples of LiF and Se are given in Figs. 6 and 7 respectively and the prominent gamma rays corresponding to ^{20}F (11.2 s) and $^{77\text{m}}\text{Se}$ (17.4 s) are marked in the Figs. Half-lives of both the isotopes were determined and Fig. 8 shows activity plot of $^{77\text{m}}\text{Se}$ (162 keV) and measured half-life was found to be

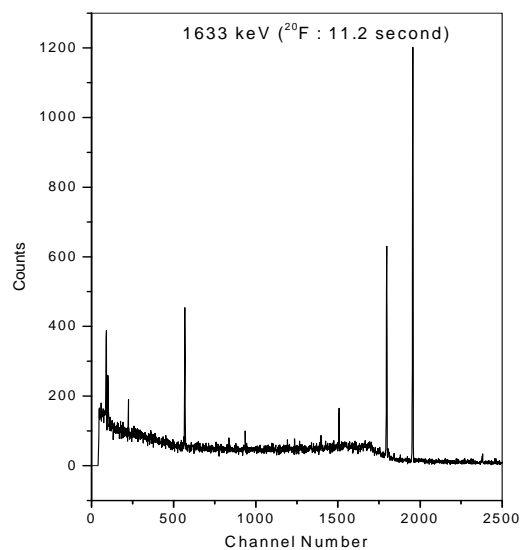


Fig. 6 Gamma ray spectrum of neutron activated fluorine standard

17.8 s, which is in good agreement with the value of 17.4 s. However, determination of these elements present in trace levels in real samples is difficult in the present set up as the rabbit can only be opened after a decay period of 1-2 minutes depending on irradiation time of 30-60 s. It is planned to make arrangements to open the rabbit with almost zero delay time to circumvent this problem. However, elements with radioisotopes having half-lives 1 min and above (e.g., ^{28}Al) are being determined regularly in the current arrangement.

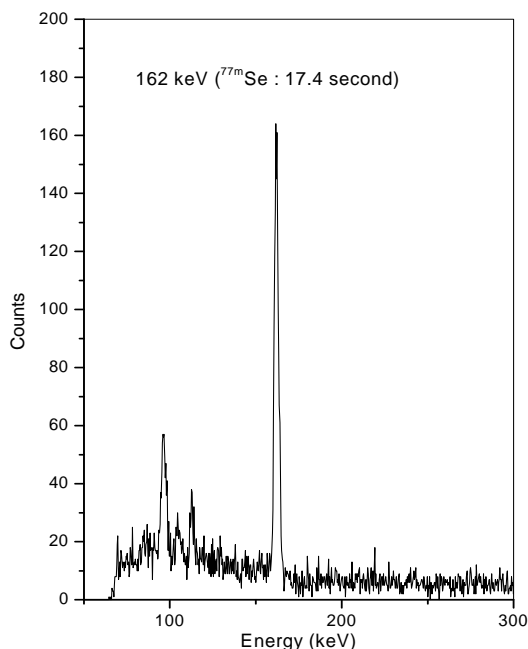


Fig. 7 Gamma ray spectrum of neutron activated selenium standard

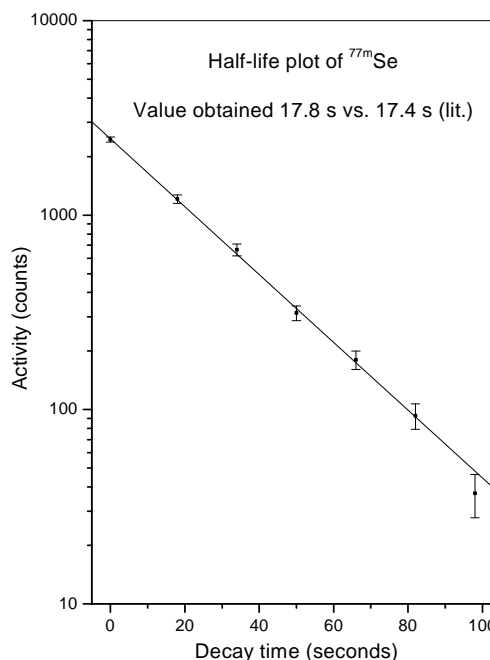


Fig. 8 Activity plot of ^{77m}Se (17.4 second)

5.5 Evaluation of enhanced sensitivities

To quantify the analytical sensitivities, two IAEA reference materials namely Lichen (IAEA-336) and freeze-dried animal blood (A-13) were analyzed using the PCF. The elemental concentrations of these reference materials were determined using k_0 -NAA method [4,9]. In this method, sample is coirradiated with a monostandard like Au. Peak areas under the characteristic gamma rays corresponding to various isotopes formed during neutron irradiation were used to calculate the concentrations of the respective isotopes (elements) present in the reference materials. Results obtained for reference material Lichen (IAEA-336) are given in Table 3 [11]. These data are average values of duplicate measurements. From Table 3, it is clear that the reported values are well within the confidence levels given in the IAEA certificates. Certain trace elements like Mg, Ti and Dy, which were not reported by

IAEA, could be detected in the present studies, essentially due to the enhanced sensitivities and possibility of measuring the samples with almost zero cooling period. It may be noted that not only short-lived isotopes but also medium-lived isotopes like ^{56}Mn (2.58h), ^{24}Na (14.595 h), ^{42}K (12.8h) and ^{82}Br (35.50 h) could reliably be measured by choosing the suitable cooling periods. This is possible only because of high neutron flux even though irradiation time is one min.

Table 2 : Details of irradiations and neutron flux values obtained at Reactor Power of 20MW (Moderator Level : 274.1 cm)

Sr. No.	Capsule No.	Weight of Au μg	Cooling Period, min.	Neutron Flux $\text{cm}^{-2}\text{s}^{-1}$ $\times 10^{13}$
1.	39	1.516	90	2.21
2.	41	1.721	85	2.08
3.	18	0.893	122	1.85
4.	42	1.439	74	2.21
5.	44	1.760	83	2.20
6.	51	1.669	67	2.01
7.	51	1.372	77	1.90
8.	52	1.617	44	1.98
9.	52	1.949	74	1.97
10.	52	1.609	47	1.90
11.	53	1.192	68	1.93
12.	54	1.197	53	1.94

The elemental sensitivities (cps/ μg) corresponding to a few short lived and medium lived isotopes, along with the similar values obtained from the measurements carried out earlier using Apsara reactor are given in Table 4. Since the samples were irradiated for longer duration (10 min) in Apsara reactor to achieve better counting statistics, the results were normalized to account for the saturation factors. The sensitivities obtained using Dhruva PCF were 10 to 100 times higher than those obtained using Apsara. Detection limits were calculated using Currie's formula [13], $L_D = 2.71 + 3.29 \sigma_b$, where L_D is the detection limit (counts) and σ_b is the square root of the background under the characteristic photo-peak of interest. L_D (counts) was converted to L_D (μg) by dividing it with sensitivity (S) in counts/ μg .

The limits of detection for many elements obtained using the PCF are better by several orders than those obtained using Apsara irradiations.

Table 3 : Some of the trace elements in reference material LICHEN (IAEA-336) analysed using Dhruva-PCF irradiations.

Element	Isotope	Half Life	γ ray Energy keV	% γ ray Abundance	Measured Concentration mg/kg	IAEA Data, mg/kg#	Confidence Level mg/kg#
Br	⁸² Br	35.50 h	554	70.9	13.75	12.9	11.2-14.6
K	⁴² K	12.36 h	1524.7	18.8	2144	1840	1640-2040
Mn	⁵⁶ Mn	2.578 h	1811	27.2	60.4	63	56-76
Na	²⁴ Na	14.959 h	1368.5	100	353	320	280-360
Al	²⁸ Al	2.241 m	1778.8	100	642	680	570-790
Cl	³⁸ Cl	37.24 m	1642	31	1792	1900	1600-2200
V	⁵² V	3.743 m	1434	100	1.34	1.47	1.25-1.69
Mg	²⁷ Mg	9.458 m	1014.4	29.1	921	NA	-
Ti	⁵¹ Ti	5.76 m	320	93	86.6	NA	-
Dy	¹⁶⁵ Dy	2.334 h	94.7	3.58	0.067	NA	-

NA : Not Available, # From Reference [12]

Table 4 : The sensitivities and the detection limits (L_D) of some elements.

Isotope	γ - ray energy keV	Sensitivity PCF, cps. μg^{-1}	L_D PCF μg	Sensitivity Apsara, cps. μg^{-1}	L_D Apsara, μg
²⁸ Al	1778	51.82	1.9×10^{-5}	1.47	5.11×10^{-3}
⁵² V	1434	147.23	8.19×10^{-6}	22.40	5.39×10^{-5}
⁵¹ Ti	320	2.67	2.81×10^{-3}	0.02	5.11×10^{-3}
²⁷ Mg	1014.4	4.98×10^{-2}	0.11	1.75×10^{-4}	31.56
³⁸ Cl	1642	9.99	1.02×10^{-4}	0.27	3.79×10^{-3}
³⁸ Cl	2167	9.76	4.61×10^{-6}	0.27	1.68×10^{-4}
⁵⁶ Mn	1811	719.60	1.12×10^{-6}	129.80	6.24×10^{-6}
²⁴ Na	1368.5	341.38	4.86×10^{-6}	2.65	6.27×10^{-4}
¹⁹⁸ Au	411.8	47742.49	1.09×10^{-7}	602.40	8.58×10^{-6}

Table 5 : Determined concentrations (in mg.kg⁻¹ unless mg.g⁻¹ is indicated) of elements in IAEA CRMs (SL-1 and Soil-7)

Element	SL-1 (Lake Sediment)				Soil-7			
	This work	Certified (info) values	Deviation %	Z-score	This work	Certified (info) values	Deviation %	Z-score
Dy	7.2 ± 0.4	7.46 ± 2.10	-3.6	-0.24	3.6 ± 0.1	3.9 ± 1.0	-8.3	-0.6
Na*	1.69 ± 0.07	1.72 ± 0.12	-1.8	-0.5	2.43 ± 0.11	(2.4)	1.2	NC
Al*	91.0 ± 3.0	(89)	2.4	NC	45.9 ± 0.8	(47)	-2.3	NC
K*	14.6 ± 0.3	(15)	-2.7	NC	12.7 ± 0.5	(12.1)	4.7	NC
Ca*	2.4 ± 0.2	(2.5)	-4.6	NC	159.1 ± 7.9	(163)	-2.5	NC
Ti*	5.23 ± 0.12	5.17 ± 0.36	1.1	0.34	3.02 ± 0.11	(3.0)	0.6	NC
V	181.4 ± 11.0	170 ± 15.3	6.3	1.5	67.0 ± 3.2	66 ± 7	1.4	0.28
Mn*	3.64 ± 0.08	3.46 ± 0.17	5.0	2.2	0.62 ± 0.02	0.63 ± 0.04	-1.6	-0.50
As	27.8 ± 1.2	27.5 ± 3.0	1.3	0.2	14.1 ± 0.6	13.4 ± 0.8	4.9	1.74
Br	7.04 ± 0.12	6.82 ± 1.7	3.1	0.26	7.5 ± 0.4	NA	NA	NC
Ba	641.0 ± 7.1	639.0 ± 5.1	0.3	0.78	117 ± 6	NA	NA	NC
Th	14.4 ± 0.7	14.0 ± 1.0	3.2	0.8	7.8 ± 0.5	8.2 ± 1.0	-5.1	-0.8
U	4.16 ± 0.21	4.02 ± 0.32	3.4	0.86	2.5 ± 0.1	2.6 ± 0.5	-4.0	-0.4

* - mg.g⁻¹, (info) – information values, NA – not available, NC- not calculated since uncertainties values of CRM are not available

Table 6 : Elemental concentrations (mg/kg unless % indicated) in NIST SRM 1633a (coal fly ash) and NIST SRM 1632a (coal)

Element	SRM 1633a (coal fly ash)		SRM 1632a (coal)	
	This work	Certified Value	This work	Certified Value
Na %	0.19±0.01	0.17±0.01	#	
Al %	15.6±0.2	(14)	2.9 ± 0.06	3.1
K %	2.00 ± 0.06	1.88±0.06	(4.1 ± 0.2) x 10 ³	4.2
Ca%	0.91±0.14	1.11±0.01	ND	NA
Ti %	0.94±0.06	(0.8)	0.19 ± 0.03	0.18
V	291.0±9.3	(300)	ND	NA
Mn	180±0.3	(190)	29.2 ± 0.2	28 ± 2
As	152.1±4.4	145±15	8.35 ± 0.03	9.3 ± 1.0
Sr	804.5±47.5	830±30	92.5 ± 7.1	NA
La	79.5±0.3	NA	13.5 ± 0.3	NA
Sm	16.8±0.1	NA	2.1 ± 0.4	NA
Dy	12.6±0.7	NA	1.9 ± 0.1	NA
Th	29.7±1.7	NA	4.6 ± 0.1	4.6 ± 0.1
U	11.8±0.3	NA	1.27 ± 0.01	2.28 ± 0.02

() – information value, NA – not available, ND – not determined, # the certified value was taken as the input for internal monostandard method.

6. APPLICATIONS

Pneumatic irradiation facility (PCF) at reactor sites, where sample is sent to irradiation position and retrieved within a short time finds many applications using NAA. PCF irradiations are useful to determine those elements, whose activation products are short-lived. It is to be understood that all the elements do not have short-lived isotopes. However, with high neutron fluxes available in the irradiation position of PCF, Dhruva and with fast retrieval times, a large number of elements that have medium and long-lived isotopes are also determined in a number of samples. After characterizing neutron spectrum, evaluating enhanced sensitivities and validating the entire procedure many samples were analyzed. In combination with long irradiations using Apsara, panoramic elemental profiles could be obtained for many samples and reference materials [14]. For example, studies of recent meteorites could be carried out [15]. Presence of various essential elements in tender wheat grass [16] and many medicinal herbs [17] was measured. Diffusion of monovalent ions like iodide in agar agar was investigated. Many nuclear materials were analyzed for impurities. Precious stones like Alexandrites [18], coal samples [19] and coal fly ash [20] were analyzed for elemental content. Leaf and soil samples from Jaduguda were analysed for toxic and heavy elements. Uranium could be estimated by using solid state nuclear track detectors (SSNTD) taking advantage of fission fragment interaction with SSNTD foil. This facility is very useful to obtain quality nuclear data. A few of them are described in the following.

6.1 *Analysis of reference materials*

For evaluating the entire methodology and to determine the accuracy of the INAA method using PCF, four reference materials namely IAEA CRMs SL-1 (Lake sediment) and Soil-7 (soil) and NIST SRMs 1633a (coal fly ash) and 1632a (coal) were analyzed. IAEA CRMs were analyzed by relative method, whereas NIST SRMs were analyzed by k_0 -based NAA. IAEA CRM SL-3 was used as the comparator in relative method as well as a control sample in k_0 -NAA. Figs. 9 and 10 are the γ -ray spectra of irradiated SL-3 corresponding to cooling periods of 3 min and 1 h. By judicious use of cooling time, 12 elements were determined. The results are given in Tables 5 and 6. For SL-1 and Soil-7, Z scores were

calculated and included in Table 6. The values of Z-score are within 1 except for V and Mn in the case of SL-1, and for As in the case of Soil-7. This indicates that our entire methodology with PCF is reliable and the results are accurate. In the case of SL-1, Al, K and Ca were determined in the present work, where IAEA gave information values. Similarly for Soil-7, 5 elements were reported with information values and two elements Br and Ba were determined for the first time. In the case of SRM 1633a, coal fly ash standard, La, Sm, Dy, Th and U are determined for the first time in this work (Table 7).

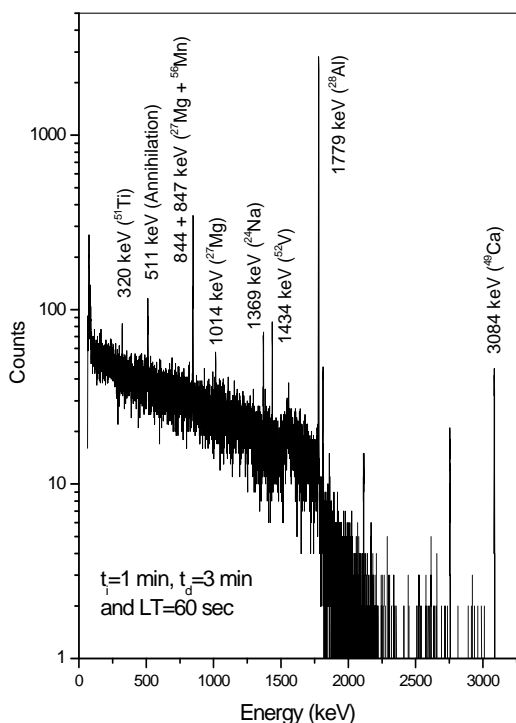


Fig. 9 Gamma ray spectrum of CRM SL-3 (Sediment)

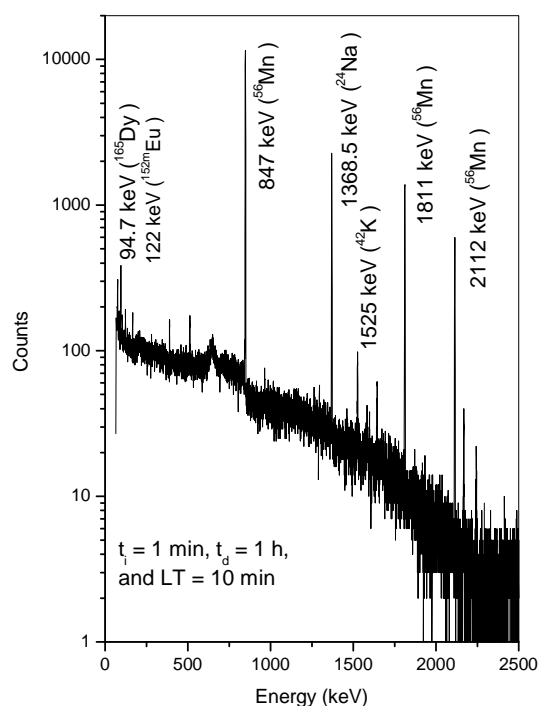


Fig. 10 Gamma ray spectrum of CRM SL-3 for medium lived nuclides using PCF

6.2 Analysis of meteorite sample

Concentrations of elements like Al, Mg, Ca and V were determined using short-lived isotopes in Jagannath meteorite [15]. The k_0 -NAA method was used for the concentration calculation. The values of the elemental ratios like Al/Ca, Al/Mg and Ca/Mg for this meteorite were found to be in good agreement with those reported for the meteorites from Kobe and Czech Republic. This confirms the authenticity of the Jagannath meteorite.

6.3 Analysis of Alexandrite samples

Four samples of Alexandrite, a variety of chrysoberyl gemstones, obtained from Visakhapatnam District, Andhra Pradesh, were analyzed for their multielement profiles by k_0 -based instrumental neutron activation analysis. The major matrix of Chrysoberyl and Alexandrite is $\text{BeO} \cdot \text{Al}_2\text{O}_3$ and they contain different elements at varying concentrations at trace levels. Chrysoberyl displays the “Alexandrite Effect” where the gemstone looks green in daylight and red in candle-light. This effect is due to the substitutions of Cr^{3+} for Al^{3+} . The Cr-containing Alexandrite appears blue-green, when viewed in daylight or fluorescent-tube lighting, and red in light from a candle or an incandescent lamp. Samples of mass about 10 mg along with a gold comparator were irradiated in pneumatic carrier facility (PCF) of Dhruva reactor. Concentrations of 13 elements including Al were determined in these samples and are given in Table 7 [18]. The accuracy of the method was evaluated using IAEA CRM SL-3.

6.4 Analysis of coal fly ash and coal samples

Samples of coal from four seams of Kothagudem coal deposit, being used in a thermal power plant at Vijayawada, and coal fly ash obtained from surroundings of thermal power plant of western Orissa, were analysed for the major, minor and trace elements using PCF, Dhruva and Apsara. NIST SRM 1633a (coal fly ash) was used as a control and concentrations of 26 elements were determined. IAEA CRM SL-3 was used as the comparator. This data was useful for a study on the trace element geochemistry of coal deposits in collaboration with Department of Geology, Andhra University [19]. Samples of mass of 10 mg were irradiated for 1 min in the PCF.

The elemental concentrations of Al, V, Ti, Mg, Ca, Dy, Mn, Na, K, As, Sr, La, U and Th, in coal fly ash, were determined from four independent experiments (Table 8) and compared with the concentrations of average crustal abundance to observe any enrichment of elements [20]. Concentrations of elements like As, La, Dy, U and Th were found to be higher than the corresponding crustal abundances.

Table 7 : Elemental concentrations (mg/kg unless g/kg is indicated) of four Alexandrite Samples.

Element	A1	A2	A3	A4
Na	216.9±5.0	133.9±2.6	161.69±3.99	134.56±2.76
Al *	330±11	403±16	407±19	396±20
K	73.3±13.6	49.88±7.52	ND	ND
Cr*	1.91±0.10	0.92±0.18	3.69±0.21	4.92±0.20
Mn	65.06±1.89	19.70±1.12	112.47±2.70	142.31±2.56
Fe*	21.9±1.4	8.1±0.7	27.4±2.0	36.2±2.1
Co	16.57±0.93	8.39±0.72	46.64±0.45	43.97±1.11
Ga	236.2±3.4	168.6±1.9	205.7±2.0	184.3±1.8
As	0.99±0.15	0.15±0.03	1.63±1.18	2.51±0.81
Br	0.23±0.02	0.13±0.01	ND	0.26±0.02
La	3.19±0.24	2.67±0.17	6.06±0.28	10.64±0.32
Ce	5.77±0.24	5.99±0.83	16.05±0.42	23.56±1.11
Sm	0.43±0.03	0.27±0.01	0.74±0.01	1.66±0.64

* g/kg, ND – not detected

Table 8 : Determined concentrations (mg/kg unless % is indicated) of elements in coal fly ash samples.

Element	Sample - 1	Sample - 2	Crustal abundance
Na%	0.067± 0.001	0.0502± 0.001	2.27
Mg%	0.67±0.05	0.52±0.04	2.764
Al %	12.3±0.2	12.0 ± 0.1	8.3
K %	1.23 ±0.01	0.82 ±0.03	1.84
Ca %	0.8 ±0.1	0.81±0.08	4.66
Ti %	1.06 ±0.05	1.02 ± 0.06	.63
V	157.2 ±8.0	151.9± 3.8	136
Mn	223.1 ± 6.5	263.6 ± 5.8	1060
As	4.24 ± 0.04	9.9 ± 0.5	1.8
Sr	240.5 ±18.0	214.8 ±18.7	384
La	128.6 ±6.3	98.4 ±5.6	35
Dy	17.3 ± 0.6	16.3 ± 0.8	4.5
Th	41.3 ±0.7	36.6± 0.2	8.1
U	10.4 ±0.1	8.4 ±0.3	2.3

6.5 Trace elements in food materials

Short irradiations with high flux are extremely useful in obtaining multi element profiles in biological samples. One minute duration irradiations in PCF were adequate to determine most of the elements in 11 varieties of neem leaves and bark samples collected in and around Roorkee. Aluminium, V and Mn were determined measuring their respective short-lived isotopes with cooling period of 3 min. When samples were measured after 15 min of cooling, many elements with isotopes having half-life in the range 30 min to 4 h could be measured. On the other hand, after cooling for a day, with long periods of counting other elements (Table 9) could be measured. As the irradiation period is short, activity levels of major matrix elements like Na and K formation was minimized. Large variations in concentrations for some elements may be attributed by authors to age difference and surroundings of the particular tree. Vanadium, Cr, Mn, Fe, Cu and Zn are of special importance in the diabetes treatment and neem leaves are used for the treatment of diabetes. Concentration ranges of these elements present in the leaves and correlations with other elements were discussed in the article [21]. In addition, chemical analysis of organic constituents gave a comprehensive understanding of the role of various trace elements and their binding with organic molecules. Similarly a large number of food materials were analysed.

6.6 Analysis of archaeological samples

Instrumental neutron activation analysis (INAA) using high-resolution gamma ray spectrometry was used to analyze five ancient pottery samples collected from excavated Buddhist sites of Andhra Pradesh, India. Sensitivities and detection limits for the elements analyzed were also calculated. Sample masses in the range of 10-12 mg were heat-sealed using polythene sheets and were irradiated for 1 min duration. A large number of elements were determined by analyzing the activities of the radioisotopes, whose half-lives are in the range of 2 min - 3 h. The results along with some results from Apsara reactor experiments are given in Table 10. For establishing the provenance/grouping of the samples of pottery, concentration ratios of Al / Sc as well as statistical cluster analysis were used [22].

Table 9 : Mean elemental concentrations in leaves and bark of Neem and SRM Peach leaves

Element	Leaves (n=11)		Bark (n=3)		Peach Leaves (SRM-1547)	
	Range	Mean±SD	Range	Mean±SD	This work	Certified/ Information
Al (mg/g)	0.14-0.36	0.25±0.06	0.21-0.68	0.40±0.20	242±15	249±7.47
As (ng/g)	131-285	190±46	104-152	120±22	63.8±1.4	60±18.0
Ba (µg/g)	22.5-42.6	33.8±10	74.9-456	203±160	112±0.02	124±4
Br (µg/g)	4.5-63.1	25.4±19.8	3.34-15.9	10.9±5.4	11.6±1.0	[11]
Ca (mg/g)	3.4-6.7	4.4±1.01	6.88-20.9	14.4±5.9	16.1±1.3	15.6±0.2
Cl (mg/g)	0.84-4.33	2.43±1.11	0.53-1.01	0.72±0.25	0.39±0.07	0.36±0.02
Co (ng/g)	36.6-118	87.5±26.9	68.4-115	85.0±18.7	62.4±4.2	[70]
Cr (µg/g)	0.47-3.31	1.34±0.95	0.96-1.18	1.06±0.08	1.03±0.03	[1.0]
Cs (ng/g)	112-204	154±31	61.9-85.2	79.3±13.3	85±2	79.7±13.6
Cu (µg/g)	2.58-8.90	6.14±1.77	2.68-5.19	3.86±1.03	-	3.7±0.4
Fe (µg/g)	86-160	122±21	143-247	186±38	195±2	218±14
Hg (ng/g)	13-34	24±5	28.0-202	84.4±71.3	29.0±0.03	31±7
K (mg/g)	11.6-35.8	20.1±7.7	1.56-2.59	2.10±0.42	23.1±0.01	24.3±1.0
Mg (mg/g)	1.25-5.21	3.56±1.19	ND	ND	3.98±0.17	4.32±0.08
Mn (µg/g)	12.0-25.8	19.2±4.8	31.5-87.6	59.2±0.6	107±2.0	98±3
Na (mg/g)	0.23-0.50	0.33±0.08	0.36-0.44	0.40±0.03	25±0.02	[24]
Ni (µg/g)	0.99-4.79	3.17±1.14	-	-	-	0.69±0.09
P (mg/g)	1.16-3.34	2.16±0.74	0.53-0.64	0.58±0.04	1.44±0.10	1.37±0.07
Pb (µg/g)	4.50-36.4	18.2±9.1	-	-	-	0.87±0.09
Rb (µg/g)	17.4-29.8	24.2±4.4	8.30-17.4	12.4±3.5	20.8±1.2	19.7±1.2
Sb (µg/g)	18.9-53.4	35.1±12.4	27.9-46.9	37.4±6.81	22.6±3.1	
Sm (µg/g)	0.09-0.23	013±0.04	-	-	0.89±0.02	[20]
Th (ng/g)	147-279	191±52	85.1-428	203±139	47±0.11	[1]
V (µg/g)	0.47-4.29	1.15±1.06	ND	ND	0.33±0.03	[50]
Zn (µg/g)	22.1-52.8	37.7±9.2	23.8-38.5	28.8±4.5	18.8±3.8	0.37±0.03

Table 10: Concentrations (mg.kg^{-1} unless mg.g^{-1} is indicated) of elements in five pottery samples

Elements	P-1	P-2	P-3	P-4	P-5
Na*	3.02 ± 0.22	3.9 ± 0.2	4.5 ± 0.2	2.31 ± 0.25	2.1 ± 0.23
Al*	78.3 ± 1.6	77.8 ± 1.4	82.2 ± 1.5	62.5 ± 1.4	68.5 ± 1.4
K*	20.6 ± 0.9	23.2 ± 0.8	18.5 ± 1.0	14.0 ± 0.8	15.2 ± 0.7
Ca*	9.1 ± 0.8	13.1 ± 1.1	9.5 ± 0.8	9.6 ± 0.9	7.6 ± 0.6
Sc	17.6 ± 0.5	12.7 ± 0.8	13.1 ± 0.8	14.5 ± 0.7	14.7 ± 0.7
Ti*	3.6 ± 0.5	4.3 ± 0.4	6.7 ± 0.4	8.2 ± 0.2	6.31 ± 0.4
V	66.7 ± 2.8	58.0 ± 3.0	88.2 ± 2.5	75.4 ± 2.6	82.9 ± 2.4
Cr	61.8 ± 2.4	66.1 ± 2.9	62.5 ± 3.0	70.0 ± 2.7	66.4 ± 2.6
Mn*	0.47 ± 0.06	0.62 ± 0.03	0.66 ± 0.03	0.62 ± 0.03	2.70 ± 0.05
Fe*	34.9 ± 0.8	37.1 ± 1.0	37.4 ± 1.0	49.0 ± 0.7	45.9 ± 0.6
Co	5.0 ± 0.3	5.8 ± 0.3	6.3 ± 0.3	6.6 ± 0.2	7.2 ± 0.3
Ga	12.0 ± 1.0	11.5 ± 0.9	11.8 ± 1.0	11.3 ± 0.9	10.8 ± 0.9
As	3.3 ± 0.2	2.6 ± 0.2	3.5 ± 0.2	6.1 ± 0.1	6.9 ± 0.1
Br	2.7 ± 0.2	2.0 ± 0.2	3.1 ± 0.3	2.0 ± 0.2	4.1 ± 0.3
Rb	162.9 ± 2.4	188.7 ± 2.2	94.4 ± 2.9	93.8 ± 2.9	85.3 ± 3.2
Cs	1.5 ± 0.1	1.7 ± 0.1	1.3 ± 0.1	1.2 ± 0.1	1.3 ± 0.1
Ba*	1.62 ± 0.01	0.54 ± 0.04	0.69 ± 0.03	0.37 ± 0.02	0.36 ± 0.02
Hf	12.8 ± 0.6	8.8 ± 0.9	9.5 ± 0.9	12.8 ± 0.6	11.6 ± 0.6
Th	18.0 ± 1.4	38.2 ± 0.8	29.3 ± 1.2	32.8 ± 1.1	25.1 ± 1.3
U	4.5 ± 0.2	3.9 ± 0.2	3.2 ± 0.2	2.9 ± 0.2	6.8 ± 0.1
Al/Sc	4449 ± 156	6126 ± 401	6275 ± 400	4310 ± 230	4660 ± 241

* - mg.g^{-1}

6.7 Analysis of Zr-Nb alloys

A non-destructive methodology was developed to determine the amount of Nb present in Zr-Nb alloys by short-lived NAA method. About 50 mg of Zr-Nb alloy samples were coirradiated with 3 mg of Nb foil of > 99.9 % purity and the activity of ^{94}Nb (6.26 min, 871.1 keV), measured three to four times in less than 30 minutes of cooling time, was used to determine the amount of Nb in the alloys with about < 2 % precision. One minute irradiation in PCF enabled the evolution of this methodology, which avoids cumbersome chemical dissolution methods. The impurity elements Al and V were also determined, though the determination of Cl and Mn required subsequent counting. Two spectra of irradiated Zr-Nb alloy acquired after cooling times of 6.9 and 16.6 min are shown in Fig. 11.

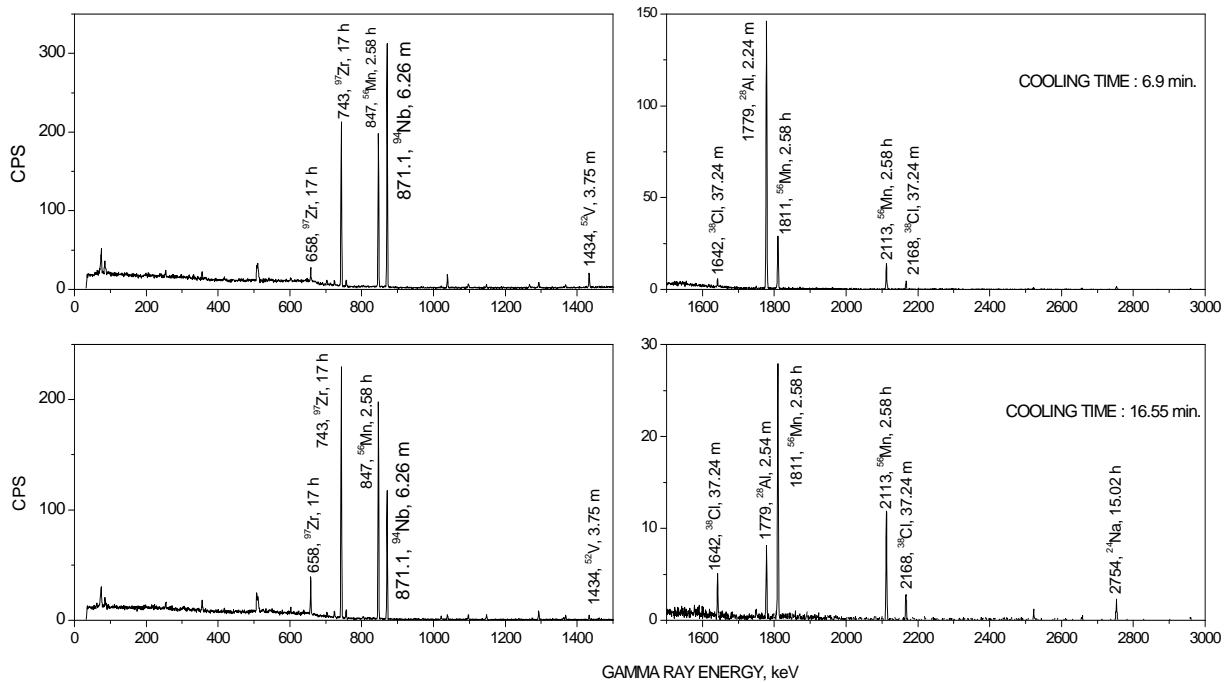


Fig. 11 GAMMA RAY SPECTRA OF Zr-Nb ALLOY, 51.1 mg, IRRADIATED IN PCF, DHRUVA, FOR 60 sec, AT 50 MW

6.8 Study of the irradiation behaviour of Dy-Ti-Oxide

Sintered sample of Dy-Ti-oxide (15.5 mg), a potential control rod material, was irradiated in PCF for 1 min. The activity of the sample was monitored using HPGe for a few hours to few days to observe the nature of the radioisotopes and their level of activation, to enable planning of schedules and adequate shielding arrangements for carrying out measurements of the long term irradiated material. This 1 min irradiation using PCF proved to be a good fore runner for the study of the irradiation behaviour avoiding high radiation dose rates. This also enabled identification and assessment of various radionuclides including the impurities. The initial measurements were done using a Pb screen of 3 mm thickness between the detector and the sample and in the later measurements the screen was removed. Bulk of the activity, as shown in Fig. 12, is due to ^{157}Dy (326.2 keV, $t_{1/2} = 8.1$ h), ^{165}Dy (94.7, 361.7, 545.8, 555.7, 633.4 and 715.3 keV, $t_{1/2} = 2.33$ h) and that of ^{153}Sm (103 keV, $t_{1/2} = 46.7$ h), as it can be seen in the spectra taken at various cooling times (Fig. 12). After few days of cooling time the radionuclide present in the sample are ^{99}Mo (140.5 and 739 keV, $t_{1/2} = 2.75$ d), ^{140}La

(328.8, 487 and 1596.5 keV, $t_{1/2} = 40.28$ h), ^{167}W (480 and 686 keV, $t_{1/2} = 23.9$ h), ^{24}Na (1369 and 2754 keV, $t_{1/2} = 15.02$ h) and ^{153}Sm (103 keV, $t_{1/2} = 46.7$ h).

6.9 Estimation of uranium by solid-state nuclear track detector

Low levels of uranium present in aqueous solutions are estimated by fission track method. An aliquot (50 μL) is transferred into a polypropylene tube, in which a small strip of lexan piece is placed and sealed. This was doubly sealed in polythene, kept in a rabbit along with gold and irradiated using PCF for 1 min duration. Fission fragments due to neutron induced fission of uranium interact with solid state nuclear track detector (SSNTD) lexan and leaves highly localized tracks. After the irradiation lexan strip is taken out, washed and alkali etched by single chemical treatment for enlarging the tracks, which are counted under a microscope. Using the track registration efficiency of the SSNTD, observed tracks are converted to number of uranium atoms by standard comparison method [23]. A detection limit of 10 $\mu\text{g.kg}^{-1}$ was achieved due to enhanced sensitivity because of highly thermalized large neutron flux in PCF, the irradiation position of Dhruva. In addition large number of samples could be analyzed that had uranium in the range of 20 $\mu\text{g.kg}^{-1}$ to 0.8 mg.kg^{-1} .

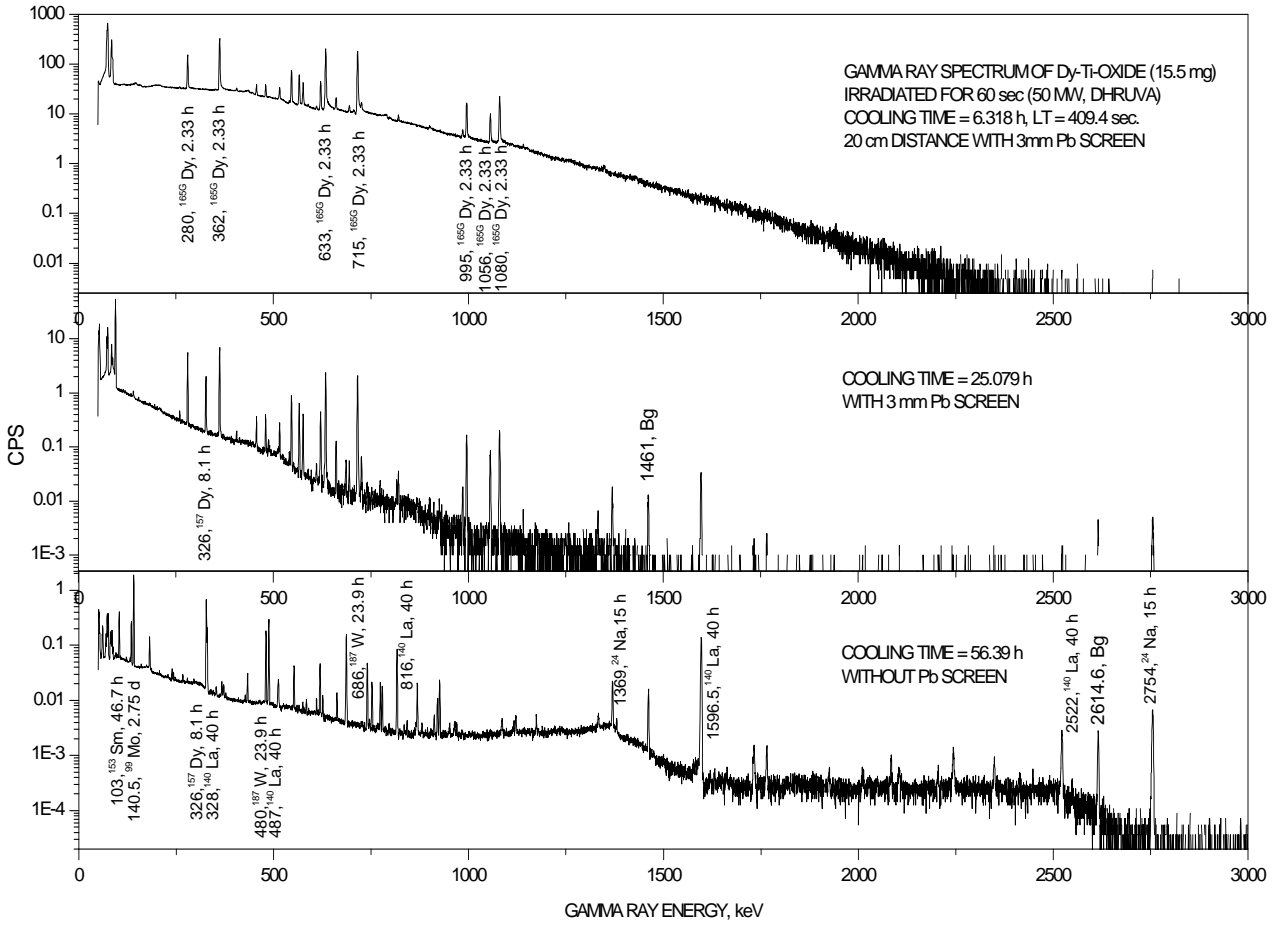


Fig. 12 GAMMA RAY SPECTRUM OF Dy-Ti-OXIDE (15.5 mg), KEPT 20 cm FROM THE DETECTOR (IRRADIATED FOR 60 sec IN PCF, DHRUVA, AT 50 MW)

7. FUTURE OUTLOOK

From the above examples it is clear that PCF is very useful for NAA. In addition, fission yields of short lived products could easily be determined although in many cases it is necessary to perform a single step fast chemical separation. As the dose rates are high due to trace level impurities, around a 3 min delay (cooling period) is required from radiological safety point of view. But the dose rate on irradiated samples is very small as the major fraction of the dose is due to rabbit, which weighs about 12 g. It is planned to make arrangements to open the rabbit by placing it in a shielded stand holder and using a long handle to unscrew the cap. Such an arrangement will enable the user under the supervision of ROD engineer and health physicist, to have access to the samples within a min. after the end of irradiation. With such a facility in place, it is possible to carry out nuclear spectroscopic studies of short-lived nuclides and analysis of samples that produce shortlived radionuclides. PCF would also provide a good opportunity, even to analyse biological and geological samples.

8. CONCLUSIONS

Neutron Spectrum in Pneumatic Carrier facility position of Dhruva reactor has been characterized by determining neutron flux and flux ratio (f -value). Neutron flux in this position was found to be around $5 \times 10^{13} \text{ cm}^{-2} \text{ s}^{-1}$ and from the f -value, the thermal component of the neutron flux was found to be $> 99.5\%$. This highly thermalized large neutron flux enables to determine those elements, which produce short-lived nuclides as well as those having low cross sections. Entire methodology was validated by re-determining the elemental concentrations in various CRMs/SRMs. The obtainable sensitivities were evaluated in comparison to versatile Apsara reactor irradiations and found that sensitivities are higher by about 100 times. Higher sensitivities and lower retrieval times of irradiated rabbit reduce turn around time. This facility is being used for analyzing various samples by NAA. Some of the samples analyzed belong to reference materials, nuclear materials, archaeology, geology, food

sciences and environment. Short irradiations and judicious variation of cooling times enabled us to determine elements that produce short and medium lived isotopes. Comprehensive elemental profiles could be obtained in combination with long irradiations using APSARA or CIRUS or DHRUVA reactors.

PCF also is used to estimate uranium by measuring the tracks created by the fission fragments in the solid state nuclear track detectors. PCF will be extremely useful for generating nuclear data on short-lived products as well as nuclear spectroscopy of short-lived isotopes.

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Annex I

PROTOCOL FOR ARRANGING PCF IRRADIATIONS

Arranging Pneumatic Carrier Facility (PCF) irradiations involve preparation of samples as per guidelines (Annex II) and a priori knowledge of major matrix elements, which is required for evaluating expected dose rates and suitability for PCF irradiations. Details shall be filled in the Pile Irradiation Request (PIR) form (Annex III) by the user and submitted to R.O.D. Office, which will be sent to Reactor Physicist for evaluation of the expected radiation dose and heat generated. Once approved for PCF irradiation, samples have to be prepared and given to ROD Personnel. They will check the suitability of rabbits using a go-no-go gauge and also arrange Health physicist to monitor radiation dose rate of the irradiated rabbit and decide to open / store the rabbit based on the dose rate.

Prior to actual irradiation, radiation measurement system has to be arranged. Users from various Divisions of BARC other than Radiochemistry Division (RCD) as well as from other units and non DAE institutions, if wish to use RCD facility, have to contact Dr. R. Acharya or Dr. A.V.R. Reddy for the availability of high resolution γ -ray spectrometer. Spectrometer has to be made on and kept ready as per Standard Operation Procedure (SOP) described in Annex IV. After the irradiations, radioactivity measurements of the irradiated samples and standards shall be made as per SOP given in Annex IV. Spectrum analysis and elemental concentrations are calculated as per SOP given in Annex VII. Record all the relevant information in the data sheets provided in the laboratory and a sample data sheet is given in Annex VIII.

Check list.

- | | | | |
|-----|---|-----|-------|
| 1. | Is PIR form filled as per Annex III ? | Y/N | _____ |
| 2. | Is it submitted to ROD ? | Y/N | _____ |
| 3. | Is the PIR approved by reactor physicist? | Y/N | _____ |
| 4. | Are the samples prepared as per guidelines ? | Y/N | _____ |
| 5. | Is the rabbit submitted to ROD ? | Y/N | _____ |
| 6. | Is the Health physicist informed ? | Y/N | _____ |
| 7. | Is the measurement system available ? | Y/N | _____ |
| 8. | Did you enter your name and other details in Register? | Y/N | _____ |
| 9. | Is the measurement system energy calibrated ? | Y/N | _____ |
| 10. | Is the detection system fit for purpose (performance) ? | Y/N | _____ |

If all the answers are Y, then and then only, irradiation SHOULD be started.

Annex II : Guidelines for Researchers for the PCF Irradiation

1. The identification number will be a four digit number preceded by two alphabets. The four digit number indicates the serial number of irradiation (in the Pneumatic Facility at DHRUVA) from the particular Unit/Division whereas the alphabets represent the research unit/division as per the following legend:

AC - Analytical Chemistry Division
RC - Radiochemistry Division
ID - Isotope Division
HP - Health Physics Division
CD - Chemistry Division
DR - Division of Radiological protection
TF - Tata Institute of Fundamental Research
e.g., RC 015 indicates that PIR received from Radiochemistry Division, BARC and Sample No. is 15
2. The identification number as indicated in (1) above shall also be written in Indelible Indian Ink on the polythene sealing wrapper of the target material. Researchers should ensure that the target material conforms to the description of material given in the PIR Form in all respects and that there is no mix-up.
3. Physical form and dimensions of the target material should be stated clearly and not by terms like “Solid” and “Metal” alone. Quantities should be expressed as milligrammes only and appropriate multiplication factors used to express gms, microgrammes etc.
4. The form should be filled in clear and legible handwriting in ink or preferably typed and submitted at least three days in advance.
5. Researcher or his representative shall be present during the irradiation.
6. Irradiations are normally carried out between 0900 h and 1600 h on working days.
7. Separate PIR form should be filled for each irradiation.
8. The irradiation requests should be approved only by officers authorized to do so as intimated to ROD by the respective Heads of the Units/Division.
9. Estimated dose rate on the sample immediately after irradiation should not be more than 10 R/h for permitting the irradiation.
10. Only polythene wrappers should be used for sealing the target materials. Other materials such as quartz and polyvinyl chloride (PVC) are not permitted.
11. More than one polythene wrappers can be irradiated in one rabbit as per one PIR form provided the Identification number on all such wrappers is same with different suffixes (For example when standards are to be irradiated along with the target material).
12. Irradiation requests, which do not conform to these guidelines have to be treated as special cases and will require clearance from Head, R.O.D.

Annex III : Application form for PCF Irradiation

**GOVERNMENT OF INDIA
BHABHA ATOMIC RESEARCH CENTRE
REACTOR OPERATIONS DIVISION
Dhruva, BARC Post Office, Bombay-400 085.**

**PILE IRRADIATION REQUEST (PIR) for Pneumatic Carrier Facility, DHRUVA
(To be submitted in triplicate to Rod Office, DHRUVA)**

Research Unit/Division: _____

Identification No. _____ P.I.R. No.: _____

(Allotted by Researcher) _____ (Allotted by Rod Office) _____

Part A: Sample details (To be filled by Researcher)

1. Chemical name and formula:
2. Physical form and Dimensions:
3. Wt, of target material _____ milligrams.
4. Stability: Stable upto _____ °C.
5. Is the material fissile/toxic: _____
6. Irradiation requested for _____ (Secs/mins) on (date) _____ at (time) _____ hrs.
7. Any other information:
Name of Researcher _____ Approved by: _____

Sign. with date: _____ Sign. with date _____

Part B: (To be filled by R.H.C. In-charge, DHRUVA only when material is fissile/toxic)

1. Special precautions:
2. Remarks: _____ Signature with date: _____

Part C: (To be filled by Safety Evaluation Section)

1. Estimated dose rate on contact, immediately after irradiation: _____ R/hr.
2. Estimated heat generation and temperature: Acceptable/Non acceptable.
3. Remarks: _____

Signature with date _____

Part D:

1. Irradiation approved/Not approved
2. Remarks: _____

(RS/ARS/Rod Engineer)

Part E : (To be filled by Officer in-charge of carrying out irradiation)

I Checklist

1. Particulate air-activity monitor is in working condition _____
2. Both wall-mounted radiation monitors are in working condition _____

Yes/No

- 3. Health Physicist present with two radiation-survey instruments _____
- 4. Ex. Fan is "ON" and cone-valve enclosure is under suction _____
- 5. Satisfactory dummy run carried out for the day _____
- 6. Identification number on sealing wrapper tallies with PIR form _____
- 7. Rabbit is from fresh stock and free from burrs _____
- 8. Rabbit checked in the dimension gauge _____

- II Irradiation details: (Reactor power _____ MW)
- 1. Shift Engineer informed and power supply 'ON' Yes/No
 - 2. Rabbit 'IN' at (time) _____ hrs. 'OUT' at (time) _____ hrs.
 - 3. Rabbit replaced at (time) (i) _____ hrs (ii) _____ hrs.
 - 4. Transit time: 1st inst. _____ (Secs), 2nd inst. _____ (Secs), 3rd inst. _____ (Secs)
 - 5. Field on contact of the cone valve immediately at the end of 1st inst. _____ (mR/hr); 2nd inst. _____ (mR/hr.), 3rd inst. _____ (mR/hr.)
 - 6. Field on completion of required time of irradiation _____ (mR/hr.)
 - 7. Shift Engineer informed and power supply put 'OFF' Yes/No
 - 8. Entries made in the register Yes/No

Name: _____ Signature with date _____

Part F: (To be filled by Health Physicist)

- 1. Contact field immediately after irradiation _____ mR/hr.
- 2. Field on shipping container (Shielding _____ inches of Pb) _____ mR/hr.

Remarks

(Signature with date)

Annex IV

STANDARD OPERATION PROCEDURE (SOP) FOR MAKING HIGH RESOLUTION γ -SPECTROMETER READY FOR MEASUREMENTS

1. Enter your name, affiliation and type of the experiment in the 'LOG BOOK' placed nearby the γ -spectrometer.
2. Ensure all electrical connections are made properly
3. Ensure that 'liquid N₂' is filled in the detector of HPGe detector.
4. Make NIM bin 'On'
5. Make PCA 'On'
6. Make a directory in the PC in your name to store your measured spectra. E.g. AVRR07. Use this directory as a temporary storage.
7. Make HV 'On' and energize the detector by increasing HV in steps of 100 V/s until the rated power is reached.
8. Ensure spectroscopic amplifier is having 'Fine gain' and 'Coarse gain' values as per the rated values.
9. Wait for 30 min before acquiring the spectrum.
10. Energy calibrate the multi-channel analyzer (MCA) as per the SOP given in Annex V
11. Clear the screen using **Clear** command key and start acquiring the background spectrum. Store this in your directory with a file name Bk-dd-mm.
12. Compare this background spectrum with the one available in the file – 'Bkgd'.
 - a. Check the integrated counts
 - b. Check the peak area under 1461 keV.

If the values are too different from the file values, contact Radiochemistry Personnel before proceeding further.

13. Determine the FWHM at 1332 keV as described in Annex VI and compare this with the value in control chart of FWHM to ensure the detection performances.
14. Calibrate the MCA for detection efficiency and obtain the efficiency curve as a function of energy. Carry out efficiency calibration as described in Annex VII.
15. Keep the actual sample in efficiency calibrated position and acquire the spectrum. Ensure that the dead time is <5%. Keep this distance between sample and the detector constant through out the measurements as much as possible.
16. After the end of measurement for a preset time, input spectrum-id like SRM-1933-PCF-01 dd-mm-yy at hh-mm. Save the spectrum as a binary file in your directory and file name should not exceed 8 digits; e.g., xyz****.bny.
17. Change the sample and repeat steps 15 and 16 until all planned measurements are completed.
18. Enter all counting details as shown in the sample given as annex VIII.
19. If the work is over, make a back-up copy of the spectra in a CD.
20. Reduce the HV of the detector to 0V in steps of 100 V/s. Put off HV and the bin power, and disconnect electrical power supply to the measuring system.
21. Enter the time in the LOG BOOK.

Annex V

STANDARD OPERATION PROCEDURE (SOP) FOR ENERGY CALIBRATION OF HIGH RESOLUTION γ -SPECTROMETER

Description: Pulse amplitude of a photo peak is a direct measure of the gamma ray energy. Response function of monoenergetic gamma rays would have a definite width depending on the detector used and the processing electronic units. Normally the shape of the response function is Gaussian and the centroid corresponds to the full energy of the gamma ray. The energy calibration procedure converts channel number of MCA into energy. Standard sources such as ^{241}Am , ^{57}Co , ^{60}Co and ^{152}Eu are used for energy calibration. This involves acquiring gamma ray spectra of standards, locating the centroid for each gamma ray, covering the entire range of interest and fitting these channel number and energy of the gamma γ -ray to a linear equation. Normally the multi channel (4k) analyzer is calibrated for 0 to 2 MeV with a slope about 0.5 keV per channel. For increasing the energy range, conversion gain and amplifier have to be changed suitably.

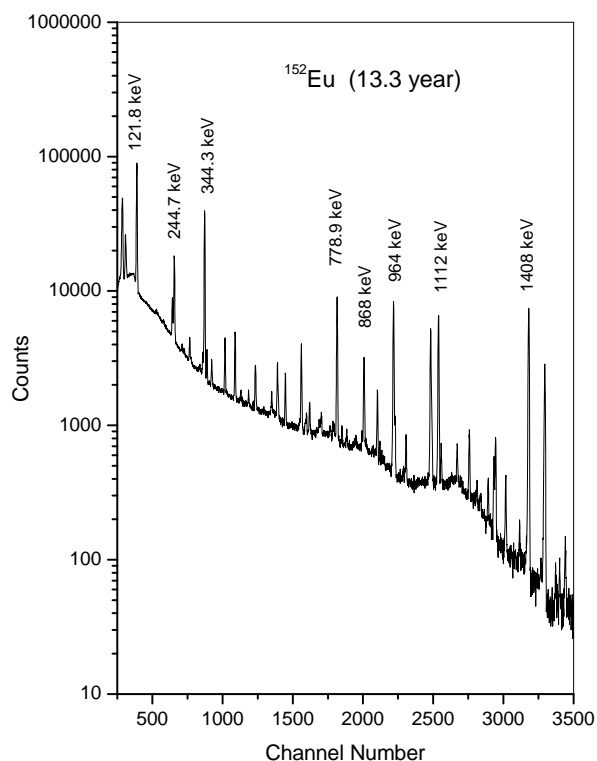


Fig. 13 A typical gamma ray spectrum of ^{152}Eu used for efficiency determination

SOP :

- a. Place ^{152}Eu γ -ray standard source in 5th shelf.
- b. Acquire the γ -spectrum (Typical spectrum is given in Fig. 13).
- c. Activate Energy Calibration.
- d. Select prominent γ -lines (121.8, 244.7, 344.3, 778.9, 1112 & 1408 keV)
- e. Find peak positions using the command **FindPeak** of PHAST software.
- f. For each centroid, enter energies using the **Enter keV** button below the spectrum window
- g. Terminate the calibration process by pressing the **Over** button. This procedure permits energy calibration with two or more points.
- h. Channel Energy Calibration plot is displayed. (First-timer should seek guidance from Radiochemistry Personnel)
- i. **Save** Calibration.
- j. Exit by pressing **Esc** key.

Annex VI

STANDARD OPERATION PROCEDURE (SOP) FOR DETERMINATION OF DETECTOR RESOLUTION

Description : Resolution of a detector is defined as its capability to resolve the signals due to two different gamma rays having close by energies. The process of conversion of absorbed energy to a signal is quite complex and the amount of energy required to produce the final signal is not constant. Thus even for the full energy absorption, the final signal has a finite spread, and this determines the intrinsic resolution of the detector. Pulse processing units further add to the spread. These fluctuations around the mean value are statistical in nature and lead to almost a Gaussian shape of the observed photo peak. The full width at half maximum (FWHM), of a peak, is a measure of the resolution R of the counting system at that energy and is given as

$$\text{Resolution (\%)} = \frac{\text{Full Width at Half Maximum (FWHM) in keV}}{\text{Peak Position (keV)}} \times 100\%$$

Any malfunction of detection system can easily be found out by change in energy resolution. The resolution of most of the HPGe detectors (16% - 40% relative efficiency) is in the range of 1.8-2.0 keV at 1332 keV of ^{60}Co .

SOP:

- a. Acquire γ -spectrum using ^{60}Co source
- b. Select R.O.I. for 1332 keV by choosing start and end position of the peak.
- c. Press Data (parameters) of peak centered gross and net peak areas, FWHM and FWTM/FWHM will appear on the screen (Fig. 14).
- d. Note down the FWHM value and include it in the control chart available in the file "Control Chart – FWHM" and typical chart is given in Fig. 15.
- e. Proceed if and only if the FWHM is within $\pm 2\sigma$ and FWTM/FWHM is 1.83-1.85, else contact Radiochemistry Personnel.

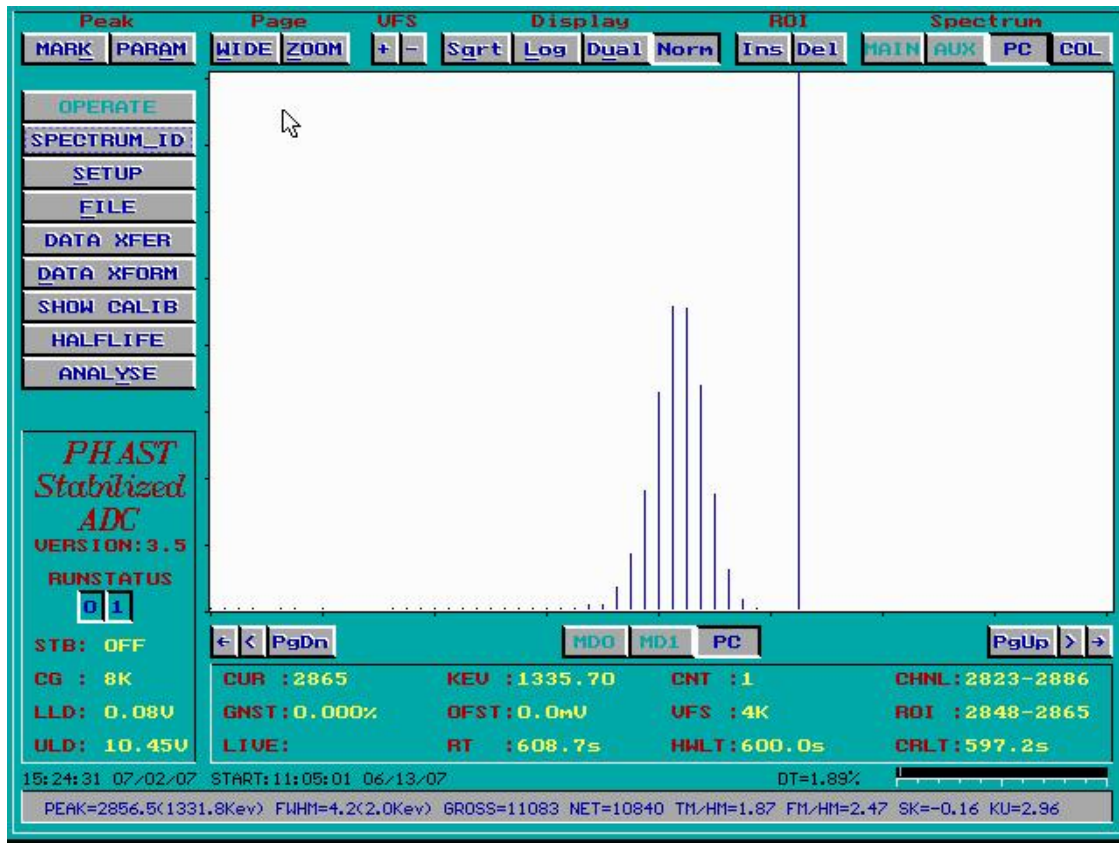


Fig. 14: A typical display a part of gamma ray spectrum wherein data on FWHM and FWTM/FWHM and peak area details of 1332 keV obtained by PHAST.

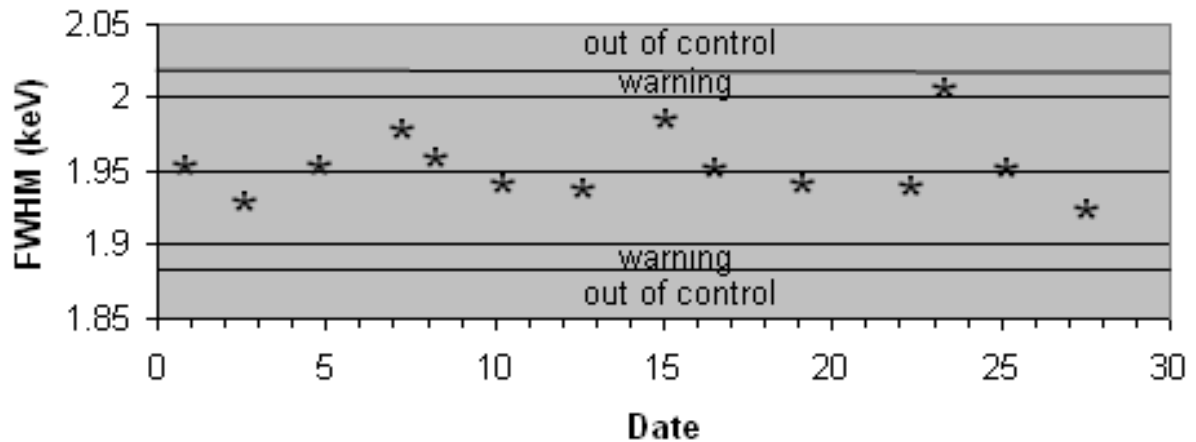


Fig. 15 : FWHM Control Chart

Annex VII

STANDARD OPERATION PROCEDURE (SOP) FOR DETERMINATION OF DETECTION EFFICIENCY

Detection efficiency is required to calculate absolute disintegration rates of radioisotopes. Detection efficiency varies as a function of gamma ray energy (E_γ). Absolute detection efficiency (ε) is determined by using primary gamma standards e.g. ^{57}Co , ^{203}Hg , ^{134}Cs , ^{54}Mn , ^{22}Na , and ^{88}Y or multi gamma ray standards e.g., ^{152}Eu , ^{133}Ba and ^{226}Ra . Choice of γ -ray sources depends upon the energy region of interest and the energies of γ -rays emitted by them. Energy ranges covered by the γ -rays of ^{133}Ba , ^{152}Eu and ^{226}Ra are 53-384 keV, 121-1408 keV (Table 11) and 186-2448 keV respectively. Efficiency calibration involves the acquisition of γ -ray spectrum of the standard source, determination of peak areas under the photo-peak corresponding to different γ -ray energies and evaluation of the detection efficiency from the measured peak areas (P_A) using the relation $\text{cps} = \text{dps} \cdot \varepsilon \cdot a_\gamma$, where cps is the count rate per second which is obtained from dividing peak area with live-time of counting of LT, dps is the disintegration rate per second of the standard source and a_γ is the gamma ray abundance. Calculated ε are fitted to the eqn. 7,

$$\ln \varepsilon(E_\gamma) = \sum_{i=0}^m a_i (\ln E_\gamma)^i \quad (7)$$

where ε is the full energy peak detection efficiency of the γ -ray of energy E_γ , a_i 's are the coefficients of the polynomial of order m . From the fitted efficiency calibration curves, constants are obtained (e.g., a_1 , a_2 and a_3 for a second order fitting) and these constants are used to calculate efficiency at any gamma ray energy. Standards for efficiency calibration in the form of weightless point sources or 5 mL liquid samples are prepared though sample geometry matching standard sources are ideal. A typical detection efficiency curve, obtained using ^{152}Eu , is given in Fig. 16.

SOP:

- a. Place ^{152}Eu γ -ray standard source in 5th shelf or the appropriate shelf of choice.
- b. Acquire the γ -spectrum (Typical spectrum is given in Fig. 13).
- c. Perform Energy Calibration of the MCA as per Annex V.
- d. Analyse the spectrum with **FindPeak** or with one of the nonlinear least square fit procedures to find the peak areas.

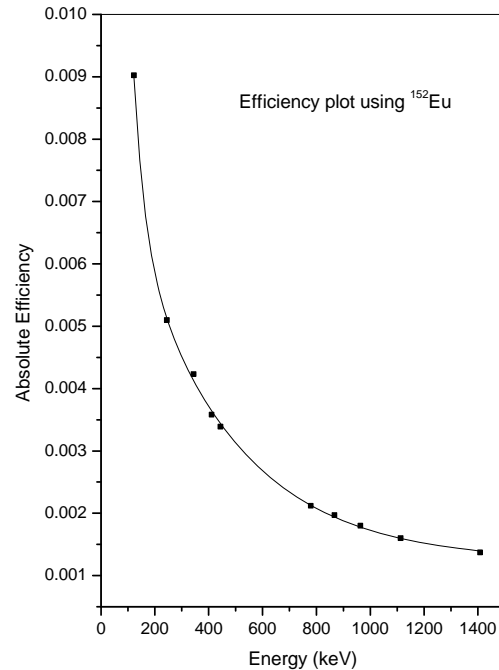


Fig. 16 Absolute efficiency of HPGe detector at 10 cm distance

- e. Mark the peaks (Table 11) and corresponding peak areas as input for efficiency calibration.
- f. Press **Effy cal** button. Typical efficiency curve (Fig 16) and coefficients for of calibration fit are displayed.
- g. Save the coefficients of efficiency calibration. These values can be used to compute efficiency at any energy up to 1500 keV. (For each shelf of the detector stand, efficiency has to be determined separately).

Table 11 : Energy and abundances for prominent gamma rays of ^{133}Ba and ^{152}Eu .

Nuclide	$T_{1/2}$	E_γ (keV)	Gamma abund. (%)
^{133}Ba	10.54 y	53.15	2.199
		81.0	34.1
		160.6	0.645
		223.2	0.45
		276.4	7.164
		302.8	18.3
		356.0	62
		383.8	8.94
^{152}Eu	13.3 y	121.8	28.21
		244.7	7.423
		344.3	26.41
		411.1	2.301
		444	3.077
		778.9	13
		867.4	4.161
		964	14.48
		1085.8	11.84
		1112.1	13.55
		1408	20.71

Annex VIII

PROFORMA TO BE FILLED BY EXPERIMENTERS

Date:

Name(s) of the user (s)

Affiliation and address

Contact number & email id

Type of the experiment

Name of sub directory

Detector : HPGe Detector No. :

High Voltage : V Amplifier Model :

Amp Coarse gain : Amp Fine gain :

Time Constant : MCA Mode : PHA/MCS

Shelf number : Calibration standard Sources : ¹⁵²Eu, ⁶⁰Co

Energy of ⁶⁰Co γ rays : 1172, 1332 keV FWHM at 1332 keV

Efficiency calibration coefts a1 a2 a3

Integrated bkg PA at 1461 keV

Table 12 : Peak areas for different gamma ray peaks of ¹⁵²Eu for efficiency calibration

Energy keV	γ abundance %	Channel No.	FWHM keV	Peak area	Efficiency
121.78	28.21				
244.69	7.423				
344.2	26.41				
778.9	13				
963.4	14.48				
1112.8	13.55				
1408.1	20.71				

