CHAPTER I

14 MeV NEUTRON GENERATOR AND ITS APPLICATION IN ACTIVATION ANALYSIS
CHAPTER 1

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1.1 INTRODUCTION TO ACTIVATION ANALYSIS:

Von Hevesy and Levi first applied artificial radioactivity to analytical chemistry in 1936\(^{(1)}\). Later on Seaborg and Livinghood\(^{(2)}\) besides demonstrating the use of the charged particles for analysis at trace levels, also indicated the non-destructive aspect of the analysis. Systematic development of activation analysis, however, only began in 1950's. Since then activation analysis is used as a very sensitive analytical tool for quantitative determination of large number of elements. Its extreme sensitivity makes it indispensable in many technical fields requiring materials of high purity. The introduction of activation analysis to medical and biological fields has led to solution of numerous, previously unapproachable problems. Activation analysis has recently found applications in criminology, forensic science, history and archaeology\(^{(3,4)}\). Apart from its high sensitivity in determination of trace elements, activation analysis also makes it possible to carry out determinations non-destructively. For this reason, activation analysis has been accepted as a method for routine elemental analysis. Activation analysis technique depends on the production of radioactive isotope of the element. This may be achieved by bombarding the sample with energetic radiations such as neutrons, charged particles or photons and subsequent measurement of induced activity. Depending upon the nature and energy of bombarding radiations, various nuclear reactions take place, which leave the nuclei either in excited state or transform them into some other nuclei with different N and/or Z. The requirement of activation analysis is that the isotope produced as a result of bombardment of irradiating particles must emit radiations which should be suitable for detection and also should be characteristic of the isotope produced. The intensity of the radiation produced by the radioactive isotope gives the amount of element present. Thus we can get qualitative and quantitative information from the
analysis of detected radiations. There are many well known chemical and spectroscopic methods used for routine analysis. The major difference between activation and these methods is that activation analysis utilizes a nuclear process, while other methods are based on atomic phenomena. Thus activation analysis gives information which does not depend on chemical composition and physical state of the element under investigation. The decision to use activation analysis is usually made by considering its advantages and limitations and also by comparing with other analytical methods, such as chemical analysis, flame photometry, X-ray fluorescence, calorimetry and atomic absorption spectrometry. Table (I) summarizes the important characteristics of above methods along with neutron activation analysis\(^5\). Generally every method has some merits and demerits, so one cannot judge the superiority of one over the other method. A detailed description of these methods, the sensitivities obtained and comparison with activation analysis is also given by Kruger\(^6\). The activation analysis offers many advantages, but the foremost advantage of this method is its sensitivity. For most of the elements, the technique is sensitive upto ppm levels which is better than atomic absorption spectroscopy. Another important advantage is the selectivity. As there are many nuclear reactions and nuclear radiations available for each element, there is large scope to avoid the interfering reactions in most of the cases. Besides these, there are many advantages of activation analysis such as 1) fast and non-destructive analysis can be done, 2) simultaneous determination of many elements, 3) the analysis is highly automatized, reducing manual errors, 4) on line analysis is possible and 5) analysis can be done regardless of chemical form of the sample. Also there is no restriction on the sample form in the sense that solid as well as liquid samples can be analyzed. Normally activation experiments are carried out either for elemental analysis or cross section measurements.

1.2 PRINCIPLE OF ACTIVATION ANALYSIS:

Activation analysis can be described in an operational sense as consisting of two major processes: (a) production of a radioactive nuclide from the desired element by some nuclear reaction and (b) measurement of characteristic radiations emitted
by the radionuclide. Thus qualitative and quantitative analysis can be performed by studying the energy spectrum and the half-life of the product nuclei.

The activity of the product nuclei \(A_0\) at the end of irradiation is given by

\[
A_0 = N \sigma \varphi (1 - e^{-\lambda t})
\]  

\[--------- (1.1)\]

where \(N\) is the number of target nuclei in the sample, \(\sigma\) is the cross section of the reaction, \(\varphi\) is the neutron flux, \(t\) is the irradiation period and \(\lambda\) is the decay constant of radioisotope.

The activity \(A_0\) of a reaction product at any time is related to counting rate \(A\) in one of its characteristic photopeaks and is given by

\[
A_0 = \frac{A \lambda}{\varepsilon \cdot f_s \cdot f_d}
\]

\[--------- (1.2)\]

where \(\varepsilon\) is the photopeak detection efficiency of the detector, \(f_s\) is the source self absorption correction and \(f_d\) is the number of gamma rays emitted per disintigration of the reaction product. When the time elapsed before start of counting and decay of radioactivity during counting period are considered, the activation equation becomes

\[
A \lambda = \varepsilon \cdot f_s \cdot f_d \cdot N \cdot \sigma \cdot \varphi (1 - e^{-\lambda t_a}) (e^{-\lambda t_a} - e^{-\lambda t_b})
\]

\[--------- (1.3)\]

where \(t_a\) is the time elapsed between end of irradiation and start of the counting, \(t_b\) is the time elapsed between end of the irradiation and end of counting. This method of analysis is known as an absolute or conventional method.

Expression (1.3) is the basic equation used in activation experiments either to calculate cross section \((\sigma)\) of the nuclear reaction or to calculate concentration \((N)\) of an element of interest in the sample.

Another approach of the radio-activation technique is based on estimation of weight of an element in a standard sample. If weight of an element in a standard sample is known, values of other parameters can be estimated comibly in the form of constant factor 'C'. For the same period of irradiation and identical conditions
of neutron flux, the weight of the unknown element in the other sample is related with the known weight of the element and is given by:

\[
\frac{W_u}{W_k} = C \frac{A^0_u}{A^0_k}
\]

Where, \(W_u\) is the weight of element in the unknown sample, \(W_k\) is the weight of the element in the standard (known) sample, \(A^0_u\) and \(A^0_k\) are the activities due to the element in the unknown and known samples respectively. The comparator method of activation analysis requires relative measurements and eliminates the need for precise nuclear data. It has therefore resulted in its more widespread use compared with absolute method.

1.3 NEUTRON SOURCES:

The neutrons, being unstable in free state can only be produced in nuclear reactions. The neutrons when used as bombarding particles offer an advantage, that they can interact directly with nuclei as the atomic coulomb field has no effect on them. Neutrons producing reactions can be induced by 1) alpha and gamma radiations from radioactive nuclides, 2) charged particle induced reactions such as (d,d) and (d,T) reactions and 3) Nuclear fission under controlled conditions.

The simplest source of neutrons is the radioisotope source which is either alpha or a gamma emitter, mixed with beryllium or other elements having low neutron binding energy. These sources are not widely used due to low neutron flux. The major disadvantage apart from low neutron flux is that these sources cannot be put off, whenever not required. The transuranic elements decay by spontaneous fission and several neutrons are liberated. Commonly the material used is \(^{252}\text{Cf}\). The neutron flux is around \(10^9\text{n/s/mg}\). The major problem faced with this source is the heat dissipation which has to be removed by cooling jackets. In accelerator neutron source the charged particles are accelerated to an appropriate energy and bombarded on a target to produce neutrons through a nuclear reaction. The reactions mostly used for production of fast neutrons are:
\[ ^{2}\text{H}(d,n)^{3}\text{He} \quad Q = 3.25 \text{ MeV} \]
\[ ^{3}\text{H}(d,n)^{4}\text{He} \quad Q = 17.6 \text{ MeV} \]
\[ ^{9}\text{Be}(d,n)^{10}\text{B} \quad Q = 3.79 \text{ MeV} \]

The advantage of the accelerators over the nuclear reactors is the moderate fabrication and maintenance costs. Further, accelerators are inherently safer, containing no dangerous amount of radioactivity, and therefore an accelerator facility to produce neutrons can be set in any laboratory or at industrial site without severe danger of radiations. Even though the sensitivity obtained in fast neutrons is not as high as that attainable in reactor activation, but still it is applicable for most of the practical applications. By \((n,p)\), \((n,\alpha)\) and \((n,2n)\) reactions, most of the elements can be detected up to ppm level.

1.4 14 MeV NEUTRON GENERATOR:

Recent developments in equipment and techniques have stimulated a great deal of interest in fast neutron activation analysis. Neutron generators appear to be attractive when analysis of large number of samples are required non-destructively. Basically neutron generator is a compact charged particle accelerator designed to produce neutrons by some appropriate nuclear reaction. Accelerator based neutron sources can provide neutrons of fairly monochromatic energy and without much technical difficulties an yield in the range of $10^8$ to $10^{10}$n/sec is achievable. Based on the capabilities of the accelerator, there can be several types of nuclear reactions suitable to produce neutrons. Amongst these $^{3}\text{H}(d,n)^{4}\text{He}$ reaction is an outstanding reaction to produce 14.7 MeV neutrons. Several reviews are available on neutron generators \(^{7,8}\). This reaction offers an advantage of producing 14.7 MeV neutrons at low deuteron energy due to high positive 'Q' value (17.6 MeV) of the reaction. The cross section of this reaction is about 5 barns at 125 KeV deuterium ion energy and therefore an ion accelerator in the energy range 100 to 150 KeV is ideal to work as a 14 MeV neutron generator. We have used the above mentioned reaction for production of neutrons. The present accelerator in the form of 14 MeV neutron
generator is used for 1) elemental analysis and 2) in measurement 14.7 MeV neutron induced reaction cross sections. The development of high resolution gamma spectroscopy and computers equally contributes for the wide acceptance of fast neutron activation analysis in diverse disciplines. The most common reactions used in activation work are \((n,2n)\), \((n,p)\) and \((n,\alpha)\).

A schematic drawing of a neutron generator is shown in figure (1.1). The neutron generator consists of following major components: i) r.f. ion source, ii) Accelerating structure with source of accelerating voltage, iii) Tritium target with cooling system and iv) vacuum system.

The basic working of the neutron generator can be summarized as follows. Deuterium ions are produced in an r.f. ion source and are accelerated in an accelerating column. The accelerating voltage is obtained from a Cockcroft-Walton voltage multiplier. The accelerated deuterium ions react with tritium and 14 MeV neutrons are generated. The reaction yield primarily depends on the intensity and purity of deuterium ion beam. To produce deuterium ions r.f. ion source similar to that of Moak\(^9\) is used. To have low gas and power consumption, a new ion source with modified geometry was designed and used in the neutron generator. The construction and working of the ion source is described in section (1.5). The flow rate of the gas inside the ion source can be controlled by adjusting the heater voltage of the thermomechanical valve. To provide high voltage of the order of 200 KV a three stage Cockcroft Walton unit is used. Rectifiers are used to charge capacitors during one half cycle and other rectifiers to transfer charge during other half cycle. The a.c. voltage in the range 5-40 KV is obtained from a step-up transformer and is fed to the multiplier circuit consisting of rectifiers and capacitors, where the rectifiers act as switches to charge the capacitors. Each capacitor is charged to twice the input peak voltage and by cascading the voltage doublers, the input voltage is multiplied three times to get required output. The high voltage measurement is made by monitoring the d.c. current flowing through a 4000 MΩ resistance connected across output. The accelerating tube consists of aluminium electrodes separated by insulating spacers of porcelain. Inside the column the electrodes are positioned in
Fig. 1.1: Schematic of Neutron Generator.
such a way that in addition to focusing and accelerating action, they help in restricting the ions from striking the walls of porcelain cylinder. With the resistance chain arrangement, equal potential difference is maintained between successive electrodes. The charged particle gains energy in relatively equal increments as it passes through the potential difference.

Power supplies for magnet coil, probe voltage, gas leak and r.f. oscillator along with deuterium gas cylinder are kept in a high voltage dome. A.c. power to all the power supplies in the dome is provided through an isolation transformer. This transformer is designed and fabricated to provide 230 V at load current of 10 A. The secondary is isolated from the primary for 300 KV by impregnating the coil in a special type of epoxy resin in vacuum.

Tritium target is one of the most important component of 14 MeV neutron generator. The commercial tritium targets are fabricated by adsorbing tritium gas in a material like zirconium or titanium which is backed by a good conductor of heat like copper or silver. The operating life of tritium target depends on following factors 1) displacement of tritium by ions, 2) diffusion of tritium in the backing material, 3) degassing of the surface due to inadequate cooling and 4) surface contamination. The tritium targets used in present work were of strength 8 Curies and were provided by isotope group, BARC Bombay. The tritium target mounted on the water cooled holder, is isolated from the ground to measure the ion current.

A clean vacuum is needed for the neutron generator, because the target being very sensitive to surface contamination causes lowering of energy and thus decreasing the neutron flux. In accelerating tube vacuum of the order of the $10^{-6}$ Torr is required to avoid scattering of ions from neutral atoms. Here we have used rotary diffusion combination with a liquid nitrogen trap, which avoids entry of oil vapors inside the accelerating tube.

The neutron generator system (figure 1.2) is kept in a concrete shielding and is operated with a control panel kept outside the shielding.
Fig. 12: Neutron generator system.
1.5 RADIO FREQUENCY ION SOURCE:

When neutrons are produced through D-T reaction, deuterium beam with high percentage of atomic ions is required. PIG and duoplasmatron type ion sources provide deuterium ion beam with high percentage of diatomic ions and therefore a magnetic analyzer is required to separate atomic ion beam. However in an r.f. ion source one can get almost 90% of atomic deuterium in the ion beam. Ideally one can achieve this using an electrodeless discharge in a glass tube. A r.f. ion source is being used in our neutron generator. Figure (1.3) shows the schematic diagram of the r.f. ion source used. The source bottle is made out of pyrex glass (recombination coefficient is of the order of 10^{-5}) tube of dimensions 12 cms long and 4 cms in diameter. One end of the tube is sealed and tungsten wire of 0.5 mm diameter and 10 mm in length was fused at the closed end. About 5 mm of this tungsten wire protruded inside the bottle. The other end of the tube is made flat. The source bottle is fitted on a aluminium flange of thickness of 10 mm, using commercial resin. The aluminium flange has one internal hole drilled connecting its periphery to its surface inside the glass bottle. Through this hole deuterium gas is fed to the ion source. The central 6 mm diameter portion of the aluminium flange acts as a first ion extraction electrode, isolated from the rest by a perspex annular disc. The second extraction electrode is made of mild steel which may be raised to negative potential with respect to ion source extraction and is normally kept at the dome potential. The efficiency of the source is increased by applying an axial magnetic field at the exit canal area. Positive voltage is applied to the tungsten electrode with respect to the aluminium extraction electrode, the latter being raised to a positive voltage in the range 1 to 5 KV with respect to the ground flange. R.f. power about 80 to 100 watts at a frequency of 40 MHz is fed to the gas inside the glass bottle through capacitance coupling. The ion source provides a steady current upto 150 μA of deuterium ions at the target and can be operated continuously for 3 hours.

1.5.1 Operation of the Ion Source:

As the r.f. field is applied to the source bottle containing the deuterium gas at low pressure the stray electrons starts oscillating with the same frequency. As
Fig. 1.3: Schematic of Ion Source.
the mean free path of the electrons in this case is much longer than the dimensions of the glass bottle, they strike the walls and release secondary electrons. Thus the electron density goes on increasing and probability of atomic collisions increases and discharge is produced. An axial magnetic field of few hundred guass is produced by a solénoid placed around the bottom part of the ion source. The presence of magnetic field together with r.f. field makes the electron path helical and increases the probability of collisions. This increases the plasma density near the extraction region. When a d.c. potential is applied to the tungsten probe, due to high conductivity plasma attains probe potential. The quartz sleeve plays an important role in extracting ions. When the probe voltage is increased, a cathode dark space develops between canal tip and plasma boundary. The quartz acts as a virtual anode and a spherical boundary is produced on the upper tip of the sleeve. The radius of curvature of the plasma boundary is determined by the potential difference, plasma density and canal to sleeve geometry. This plasma boundary acts as ion emitting surface. As the probe voltage is slowly increased, the field gets shaped such that the ions removed from the plasma boundary get accelerated towards the tip. As ions come nearer to the tip their energy increases and they follow a less curved path and come out of the canal. The height of the quartz above the canal tip and its alignment is very important as they shape the field.

1.5.2 Operating Characteristics of Ion Source:

Before installation of the ion source on neutron generator bench tests were conducted to get optimum operating conditions. The of the bench test set up is as shown in figure(1.4). The intensity of deuterium ion beam depends on parameters such as 1) extraction voltage, 2) r.f. power, 3) gas pressure, 4) magnetic field and 5) canal geometry. To study the influence of one parameter on ion current, other parameters were kept constant. On the basis of original work of Moak et al\(^{(9)}\), a large number of research papers have been published on r.f. source but almost all the designs are based on empirical rules. This is probably due to the fact that too many interdependent parameters are involved. Therefore usually it is simpler to make an ion source and vary the parameters one by one to find out optimum setting.
Fig. 1.4: Bench test setup.
for each parameter. The r.f. power (100 watts and 40 MHz) was fed to the plasma using an oscillator. The load impedance consisted of a 3" diameter coil (2 turns) of 3/16" O.D. copper tubing and the capacitor formed by the copper clips connected to the glass bottle. Capacitive coupling is preferred over inductive coupling, since at this frequency power transferred is more compared to other types of couplings. While fabricating the ion source system, two precautions were taken. First the alignment of the ion extraction system was carefully studied and the canal was fixed exactly at 90° to the flange surface. Secondly the gas feed system was completely made leak proof. For good alignment the aluminium canals were machined in one setting to avoid eccentricity. A number of canals were made out of pure aluminium rod. All the canals were of the same length but of different hole diameters. Similarly canals with different lengths but of same diameter were also made. Sixteen canals with different canal lengths (l = 7 mm, 9 mm, 11 mm and 13 mm) and diameters (d = 1 mm, 1.5 mm, 2.0 mm and 2.5 mm) were fabricated. The canals were made in screw form so that they can be fixed to any desired position externally. Also this arrangement gives one extra advantage of variation of height of silica tube over the canal tip by simply moving the canal to the desired depth. The height of quartz sleeve above aluminium canal is very critical. It was observed that when this height is equal to aluminium tip diameter maximum ion current was obtained. Each canal was fitted to the source and tested independently. Ion current was measured using a re-extractant type faraday cup. Graphite was used as a collecting electrode because it has very small secondary electron emission coefficient.

The performance of the ion source was tested by measuring the ion current with the variation of all the parameters such as probe voltage, r.f. power, magnetic field and gas pressure. The usual way, which is also adopted here is to vary the above parameters one by one keeping all other parameters constant. By knowing this one can know the optimum operating conditions of the ion source.

In the first set, the dependence of ion current on magnetic field was studied. The magnetic field was varied in five steps by monitoring the current flowing through the solenoid keeping the r.f. power and gas pressure constant. The variation of ion
current with extraction voltage at different magnetic current was found. The results with one of the extraction canals is shown in figure (1.5). It was found that ion current increases with increase in magnetic field. The magnetic field provides spiral motion to the electrons and thereby increases degree of ionization and therefore the increase in current tends to saturate after certain value of magnetic field. The second set of observations were made to measure ion current for different values of r.f. power, keeping all other parameters constant. The output power was varied by varying the plate voltage of the oscillator. The increase in power increases the output ion current. The results for particular value of magnetic field, canal length and diameter and gas pressure are as shown in figure (1.6).

It is well known that for extracting maximum current the electric field and the ion density on the plasma boundary play an important role. The initial beam convergence is determined by the concave shape of the plasma boundary, which is decided by the ion density and the electric field. The ion density can either be increased by increasing the r.f. power, or gas pressure or magnetic field. Due to space charge repulsive forces become predominant and as a result concavity of the plasma boundary decreases with increase in the ion density. This tends to increase the beam diameter and a larger portion of the beam is lost to the extracting canal. This is clearly seen in figure (1.5), where the plasma density is increased due to increase in the magnetic field and has resulted in less output current. It has been observed that the ion current also depends on the height of quartz sleeve over the canal tip. The intensity of ion current was found to be maximum when the height of quartz sleeve was nearly equal to the diameter of the canal. After optimizing all the operating parameters, the variation of ion current with different canal lengths and different canal diameters was studied and the results are shown in figures 1.7 and 1.8 respectively. It was found that with increase in canal length the ion current decreases. This may be due to the fact that ions bombard on the canal wall and loose their tracks. The ion current was found to be relatively more for large diameter of the canal since more ions get transported to the active region. However, large diameter of canal (>4mm), it was difficult to maintain a constant gas pressure in
Fig. 1.5: Variation of ion current with extraction voltage.
Fig. 16: Variation of ion current with extraction voltage for various values of R.F. power.
Fig. 1.7: Variation of ion current with extraction voltage for different canal lengths.

CANAL DIAMETER: 1.5 mm
GAS PRESSURE: 5 x 10^3 TORR
CANAL LENGTH: 1 mm
MAGNET CURRENT: 5 A
Fig. 1.8: Variation of ion current with extraction voltage for different canal diameters.
the ion source, and therefore an optimum value of canal diameter was fixed, which was 1.5 mm.

The bench test of the ion source proved to be informative in choosing suitable extraction geometry. To obtain a stable ion current, various parameters can be easily adjusted with the help of this study. The typical optimised parameters of the ion source are as follows a) canal length 9 mm, b) canal diameter 1.5 mm, c) extraction voltage 3.5 KV, d) r.f. power 100 Watt, and e) magnet coil current 5 ampere. A maximum current, of 150 μA was obtained using this set of parameters.

1.6 ACTIVITY MEASUREMENTS:

For measurement of energies and intensities of gamma rays use of a 3" × 3" NaI (TI) scintillation detector and a coaxial HPGe detector with 38% relative efficiency was made. Both the detectors were coupled to PC based multichannel analyzer. Use of standard computer programme was made for the analysis of the stored data.

The measured value of the resolution of HPGe detector is 2.5 KeV at 1332 KeV, while that of NaI(TI) detector it is 7.6 % at 662 KeV. In order to reduce the background activity, the detectors were kept in lead castles with copper lining. Efficiency of both the detectors was determined for different energies using standard gamma ray sources. Figures (1.9 and 1.10) shows efficiency calibration curves for HPGe and NaI(TI) detectors respectively. These efficiency curves were used in analysis of all the gamma ray spectrum.
Fig. 1.9: Efficiency - energy curve for HPGe detector.
Fig. 1.10: Efficiency-energy curve for NaI(Tl) detector.
<table>
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<tr>
<th></th>
<th>Atomic emission plasma spectrometry</th>
<th>Atomic absorption spectrometry</th>
<th>Atomic fluorescence spectrometry</th>
<th>X-ray fluorescence spectrometry</th>
<th>Spark source spectrometry</th>
<th>Neutron activation analysis</th>
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