EXPERIMENTS IN RADIOCHEMISTRY

D.D. Sood

S.B. Manohar

A. V. R. Reddy

INDIAN ASSOCIATION OF NUCLEAR CHEMISTS AND ALLIED SCIENTISTS

Printed by : Sharda Stationery & Xerox
Shop No. 4, Arjun Centre,
Govandi East,
Mumbai - 400088
Tel. : 9869004377

First Edition : 1994
Second Edition : 2005
Second Edition (rev): 2013
Third Edition : 2014
Reprinted : 2022

For copies and other information, please write to:
Secretary,
Indian Association of Nuclear Chemists and Allied Scientists
C/o Radiochemistry Division
Bhabha Atomic Research Centre
Trombay, Mumbai 400 085
INDIA
FAX: +91 22 25505151

About IANCAS

The Indian Association of Nuclear Chemists and Allied Scientists (IANCAS) was founded in 1981 with an objective of popularizing nuclear sciences among the scientific community in the country. Under its mandate, IANCAS is continuously promoting the subject of nuclear and radiochemistry, and use of radioisotopes & radiation sources in education, research, agriculture, medicine and industry by organizing seminars, workshops and publishing periodical thematic bulletins. With its enthusiastic Life Members from all over the country and overseas, IANCAS has become one of the popular platforms for popularizing the subject of nuclear and radiochemistry across the country. The Association's activities can be seen on its website: www.iancas.org.in.

IANCAS brings out quarterly thematic bulletins on the topic of relevance to the nuclear science and technology with the financial support from BRNS. The Association's popular book on "***Fundamentals of Nuclear and Radiochemistry***", "***Introduction to Radiochemistry***" and "***Experiments in Radiochemistry"*** is widely sought amongst the academia, researchers and students of DAE, non-DAE units and Universities. Another popular book of INCAS is "***Nuclear Materials***" which has been translated into Arabic, Hindi, Marathi and Telugu. Till December 2022, IANCAS has published 78 Thematic Bulletins and several special Bulletins. All IANCAS Books and Bulletins are available for free download on its website: www.iancas.org.in.

As a part of outreach programs, IANCAS conducts workshops on "*Radiochemistry and Applications of Radioisotopes*" at various universities, institutes, schools and colleges. These workshops play paramount role in motivating the young researchers and encourage them to accept the subject of radiochemistry and applications of radioisotopes. With the support from BRNS, a set of G.M. Counter / NaI(Tl) detector(s) are donated to the host institute of each National Workshops for their academic use. Similarly, IANCAS is also conducting one-day school Workshops to encourage and popularize the nuclear and radiochemistry subject amongst the young students. These workshops are conducted by the IANCAS main body and its four regional chapters (Southern Regional Chapter at Kalpakkam, Tarapur Chapter, Northern Regional Chapter at Amritsar, and Eastern Regional Chapter Bhubaneswar) covering the entire nation.

IANCAS is the co-organizer of biennial Symposia on Nuclear and Radiochemistry, popularly known as NUCAR. NUCAR series of Symposia covers the scope of nuclear chemistry, nuclear probes, chemistry of actinides, fission and activation products, nuclear and radioanalytical techniques, applications of radioisotopes in medical, agriculture and industries, nuclear safeguards, NUMAC & nuclear forensics, and nuclear instrumentation. In its series, the 16th biennial NUCAR-2023 Symposium will be organized at DAE Convention Centre, Anushakti Nagar, Mumbai in May 2023.

To encourage scientists / researchers actively pursuing activities in this discipline, IANCAS has instituted several awards. ***Dr. M.V. Ramaniah Memorial Award*** is conferred annually to an outstanding scientist for their significant contributions and lifetime achievements in the field of nuclear and radiochemistry. **Dr*. Tarun Datta Memorial Award*** is given annually to a young scientist (below 45 years of age) with minimum 5 years of research experience in the field of nuclear and radiochemistry and applications of radioisotopes. IANCAS also gives ***Prof. H.J. Arnikar Best Thesis Award*** annually for the Ph.D. research in the nuclear and radiochemistry area. Additionally, IANCAS also gives about 20 best paper awards to young researchers for presetting their work in NUCAR symposia.

To achieve the excellent in its cause for popularizing the nuclear sciences through electronic media, IANCAS has its own website (www.iancas.org.in) which is updated regularly. Information about the workshops, Awards and various activities of IANCAS are available on the website. All the publications of IANCAS including bulletins and books are available in free downloadable form.

Seraj A. Ansari
General Secretary, IANCAS
September 5, 2022

CONTENTS

APPENDICES

Chapter 1

INTRODUCTION TO RADIOCHEMISTRY

1. BASIC ASPECTS OF RADIOCHEMISTRY

1.1 Introduction to the Nucleus

Robert Boyle developed the technique of chemical analysis and, in 1661 AD, proposed that an element was a material that cannot be broken down into simpler substances. The postulate has been refined over the years and the smallest chemically identifiable constituent of matter is called an atom. The discovery of electrical nature of matter (e.g. Allesandro Volta, Micheal Faraday), X-rays (Conrad Roentgen), and radioactivity (Henry Becquerel) opened up a new area of research in chemistry and physics, and led to the discovery of the building blocks of an atom, namely electron, proton and neutron. Electrons have unit negative charge, protons have unit positive charge and neutrons do not have charge. Mass of a neutron or a proton is nearly equal to 1 atomic mass unit (amu) and 1 amu = 1.667×10^{-27} kg[1] (Appendix I). The mass of an electron is about 5.485×10^{-4} amu. The sum of the number of protons (Z) and neutrons (N) is called the mass number (A). The diameter of an atom is of the order of 10^{-10} m. The structure of the atom, as we know today, was elucidated by the work of a large number of scientists, the prominent among them being J. J. Thomson, Ernest Rutherford, and Neils Bohr. The central core of the atom is called nucleus. Nucleus consists of protons and neutrons and both are called nucleons. Neutrons and protons account, essentially, for all the mass of an atom. Diameter of a nucleus is about 10^{-14} m. The electron cloud occupies the vast space around the nucleus in well defined orbits. Atom is electrically neutral and the number of protons (Z) in its nucleus is equal to the number of electrons (Z). Z is known as atomic number and, characterizes an element and its place in the periodic table.

The nuclear volume is small ($\sim 10^{-41}$ m^3) and density of the nucleus is very high ($\sim 10^{17}$ kg m^{-3}) as most of the mass of the atom is concentrated in the nucleus. Therefore nuclear forces, that hold nucleons together, are very strong. Nuclear forces are stronger than the gravitational and electromagnetic forces by many orders of magnitude. They are also attractive and short ranged.

For an element with atomic number Z, the number of neutrons (N) can be different, and there is a corresponding variation in the mass number (A). Nuclides having the same Z but different mass numbers are called isotopes of an element; e.g. hydrogen has three isotopes with Z = 1 but the values of A are 1, 2 and 3. An isotope is represented as $^{A}_{Z}X_{N}$ where X is the chemical symbol e.g. $^{23}_{11}\mathrm{Na}$, $^{40}_{20}\mathrm{Ca}$ and $^{135}_{53}\mathrm{I}$ are some typical isotopes. Often Z is dropped from this representation since the chemical symbol defines the value of Z and accordingly ^{22}Na, ^{40}Ca and ^{135}I represent respectively the above isotopes.

1.2 Stable and Unstable Nuclides

There are about 275 stable isotopes present in nature. Naturally occurring uranium and thorium, have only long lived radioactive isotopes. Most of the products in the decay chain of these heavy elements are also radioactive. Radioisotopes like ^{3}H and ^{14}C are being continuously formed by cosmic rays induced nuclear reactions and are present in nature. In addition to these naturally occurring radioisotopes, a large number of radioisotopes are produced artificially by nuclear reactions. Stability of the nucleus can be understood by considering the energy which is released when neutrons and protons combine to form the nucleus. Binding energy (B) is the energy liberated when Z Protons and N neutrons combine to form a nucleus. Conversely it is the minimum energy that must be supplied to break the nucleus into its constituent nucleons[1]. Quantitatively it is equal to the difference between the mass of an atom (M amu) and sum of the masses of N neutrons, Z Protons and Z electrons of that atom, and is given by eqn. (1.1)

$$B = [ZM_H+NM_n-M] \text{ x } 931.494 \text{ MeV} \quad (1.1)$$

where M_H and M_n are masses of hydrogen atom (1 proton + 1 electron) and neutron respectively in amu and mass of electrons is neglected. Binding energy per nucleon (B/A), known as average binding energy, is a convenient quantity to compare the stability of different nuclides or isotopes. Greater the average binding energy, more stable is the nucleus. For example, B/A of ^{40}Ca and ^{41}Ca are 8.551 and 8.547 MeV, respectively indicating that ^{40}Ca is more stable than ^{41}Ca. The average binding energies of $^{135}_{52}$Te, $^{135}_{53}$I, $^{135}_{54}$Xe and $^{135}_{55}Cs$ are 8.345, 8.385, 8.399 and 8.402 MeV, respectively. This indicates that stability of these nuclides are in the order ^{135} Te < ^{135}I < ^{135}Xe < ^{135}Cs. In fact, the first three are short lived radioisotopes, whereas ^{135}Cs half life is $2\text{x}10^6$ years.

1.3 Radioactive Decay

Unstable nuclides (radioisotopes) disintegrate (decay) by emitting ionizing radiations, α, β and γ. Disintegration (radioisotopes) process eventually leads to a stable end product. In α-decay, an alpha particle, equivalent to helium nucleus, is emitted and the mass number of the product decreases by four units and atomic number decreases by two units, e.g.

$$^{238}_{92}\text{U} \rightarrow {}^{234}_{90}\text{Th} + \alpha \quad (1.2)$$

In the β-decay a bound neutron is converted to a bound proton, an electron and an antineutrino ($\bar{\nu}$) are emitted. In the β^+ and EC decay, a bound proton is converted to a bound neutron, and a positron (β^+) and a neutrino are emitted. On the other hand in EC, a bound electron is captured by a nucleus and a neutrino (ν) is emitted. In the β^+, β^- or EC decay, atomic number changes by one unit and mass number remains constant. All these decay processes are called beta decay processes. Examples for each decay process are given below:

$$^{132}_{52}\text{Te} \rightarrow {}^{132}_{53}\text{I} + \beta^- + \bar{\nu} \quad (1.3)$$

$$^{11}_{6}\text{C} \rightarrow {}^{11}_{5}\text{B} + \beta^+ + \nu \quad (1.4)$$

$$^{61}_{30}\text{Zn} + \text{e} \rightarrow {}^{61}_{29}\text{Cu} + \nu \quad (1.5)$$

In the β-decay, one of the bound neutrons of the nucleus is converted to a bound proton as follows:

$$n \rightarrow p + \beta^- + \bar{\nu} \quad (1.6)$$

whereas in β^+ decay one bound proton is converted to a bound neutron as follows:

$$\text{p} \rightarrow \text{n} + \beta^+ + \nu \quad (1.7)$$

---

[1] Study of the properties of unstable nuclides gives an insight into many nuclear properties, but these are not described in this book

Electron capture (EC) competes with β^+ decay as a possible alternative. In this process an orbital electron is captured by the nucleus resulting in the transformation of a bound proton to a bound neutron.

In a majority of cases, α and β decay processes produce a daughter nuclide in an excited state. Subsequent de-excitation is normally accompanied by gamma ray emission due to transition between two nuclear levels. The emitted γ-rays thus have discrete energy characteristic of the daughter nucleus. Most commonly this de-excitation occurs in a very short period (few femto seconds). However, in a few cases there is a measurable half life ranging from microseconds or more. Such nuclides are called isomers and the transition is called isomeric transition (IT). An example of isomeric transition is:

$$^{131m}Te^* \rightarrow {}^{131}Te + \gamma \qquad (1.8)$$

Gamma ray emission also occurs during many nuclear reactions. One of the possible decay modes of heavy nuclides like ^{252}Cf, ^{244}Cm and ^{238}U, is the spontaneous fission (SF). In SF, the nucleus breaks into two fragments of comparable mass, as shown below:

$$^{252}_{98}Cf \rightarrow {}^{146}_{56}Ba + {}^{106}_{42}Mo \qquad (1.9)$$

1.3.1 Radioactivity

Radioactivity (A) of a radioisotope, expressed as number of disintegrations per unit time, is equal to the product of the number of atoms (N) of the decaying radioisotope present and its decay constant (λ). Thus

$$A = -\frac{dN}{dt} = N\lambda \qquad (1.10)$$

The negative sign indicates the decay of the radioactive nuclide which results in the decrease of the number of atoms with time due to transformation to a daughter product. Decay constant λ is characteristic of a radioisotope. The decay rate depends only on the number of atoms of a radioisotope in the sample.

1.3.2 Half-life

Radioactive decay follows first order kinetics as the rate of the decay depends only on the number of atoms (N) of a radioisotope present in the sample. Units of radioactivity are Becquerel (Bq) and Curie (Ci). 1 Bq = 1 dps (disintegrations per second) and 1 Ci = 3.7×10^{10} dps. The half-life ($t_{1/2}$) of a radioisotope is defined as the time interval in which the number of atoms of the original radioisotope reduces to half of the number initially present ($N = N_0/2$). As the decay is a transformation process, an equal number of product atoms are formed. λ and $t_{1/2}$ are inversely related as $t_{1/2} = \ln 2/\lambda = 0.693/\lambda$. Radioactivity (A) is often referred to as activity. Experimentally activity is measured in counts (c) per unit time[2]. The value of C is dependent on the detection efficiency of the counting system for a specific source-detector geometry. The activity (A) of a radioisotope at any given time (t) is given by

$$A = A_0 e^{-\lambda t} \qquad (1.11)$$

where A_0 is the activity initially present (t = 0). A typical decay plot of ^{32}P is given in Fig.1.1, where A is plotted as a function of time. By plotting log A as a function of time, a straight-

---

[2] A is also defined in units of disintegration per unit time

line will be obtained whose slope is equal to 2.303 λ from which $t_{1/2}$ of that radioactive nuclide is calculated.

1.4 Properties of Nuclear Radiations

In the study of radioactivity, knowledge of three parameters is necessary. These are (i) types of radiations emitted, (ii) energies of these radiations and (iii) the number of radiations emitted per unit time. Radiation is detected by measuring the effects produced by its interaction with matter. It is, therefore, essential to understand the interaction of radiation with matter. These interactions depend on charge, mass and energy of the radiations as well as the properties of the materials with which they are interacting. Energies of α rays emitted in the decay of naturally occurring radioisotopes vary from 4 to 8 MeV and their range in air is 2 to 3 cm. A thin paper can stop α rays. Energies of beta rays vary from a few keV to 4 MeV and they can be stopped with aluminium foils. The thickness of the foil required for stopping β rays depend upon their energy. β^+ is anti particle to electron and gets annihilated by combining with an electron in the medium. X- and γ-rays are electromagnetic radiations and thick lead sheets are needed to stop them.

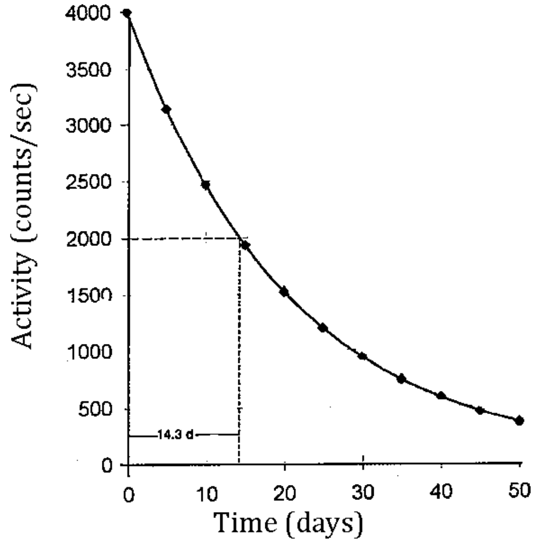


Fig. 1.1 Radioactivity (A) profile of ^{32}P as a function of time (t)

1.5 Interaction of Radiation with Matter

The passage of radiation through matter results in the deposition of its full/partial energy in the medium traversed by the radiation and, may cause ionization and excitation of atoms and molecules. If the ionization/excitation is measured, then it serves as the primary signal to detect the presence of radiation and often its quantitative measurement. Nuclear radiations are detectable only through their interaction with matter. Radiation detection, therefore, depends on the manner in which the radiation interacts with the material of the detector. For measurement and characterization of nuclear radiations, it is necessary to understand the interaction of various radiations with matter through which they are passing.

Nuclear radiations are broadly classified into two categories: (i) charged particulate radiations, e.g., p, α, fission products, β^- and β^+, and (ii) uncharged radiations, e.g., n and γ. Charged particulate radiations interact with the electrons of the medium through coulombic forces and lose their energy. On the other hand, uncharged radiations undergo interaction in two steps or they pass through matter without interacting. Charged particles continuously lose energy while passing through a medium due to coulombic interaction with electrons in the medium. Contrary to the charged particles, a γ-ray may lose its entire energy in a single collision with an atom of the medium (photoelectric effect or pair production) or it may undergo compton scattering with the electrons in the medium and partially lose its energy. Neutrons lose energy by elastic and inelastic collisions or may be captured by nuclei of atoms in the medium.

2. INTRODUCTION TO RADIATION DETECTION

Radiation detection methods are based on the measurement of the charge produced due to interaction of radiation with the detector material. The net result of the radiation interaction is the appearance of electric charge within the active volume of the detector[3]. The interaction times are so short[4] that the deposition of radiation energy can be considered as instantaneous and charge appears in the detector at zero time. This charge must be collected to form the primary signal for radiation detection. Charge is collected by applying an electric field across the detector. Time required for charge collection depends on the mobility of charge carriers in the detector[5] and the average distance they travel. The charge collected is so small that the signal has to be amplified before any measurement can be made. After amplification the signal is further processed to obtain intensity (total number) of radiations of all energies (gross counting), or intensity as a function of energy (spectrometry).

Detectors are operated either in current mode or pulse mode. In pulse mode operation, interaction of each individual quantum of radiation is recorded. Thus in pulse mode the information on the energy of each radiation can be obtained. In the current mode, average current produced as a result of interaction of radiation, is measured. It depends on the product of interaction rate and the charge produced per interaction. Thus in this mode, the average event rate and charge produced per event are obtained. This mode of measurement is very useful where the event rate is high, e.g. in dosimetry.

Over the years, a variety of radiation detectors are developed. They can be classified based on the detector material used and/or type of measurement that can be made, e.g., gas filled detectors in which ionization produced in the gas is measured. Some materials absorb radiation energy and produce scintillation that can be measured. Yet another class of detectors are based on using semiconductor materials like Ge and Si. Detectors filled with gases like BF_3, ^3He and ^4He are extensively used for neutron detection.

When the detection system is used to get information on the number of disintegrations only, then it is called gross counting system. A simplified block diagram of such a system is given in Fig. 1.2. If the detection system is used to derive the information on intensity as a function of radiation energy, then the measurements are called spectrometric measurements. A block diagram of a spectrometric system is given in Fig. 1.3.

---

[3] This is true for gas based and semiconductor detectors. For scintillation detectors, the emitted light in the detector is converted to charge and multiplied in a photomultiplier tube.

[4] Interaction or stopping time is about a few nanoseconds in gases and a few picoseconds in solids.

[5] Charge collection time in gas based detectors can be as long as a few milliseconds whereas in semiconductors it is a few nanoseconds

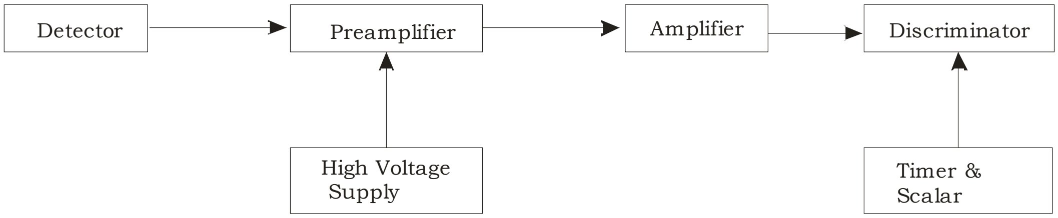


Fig. 1.2 Block diagram of simple system for gross counting

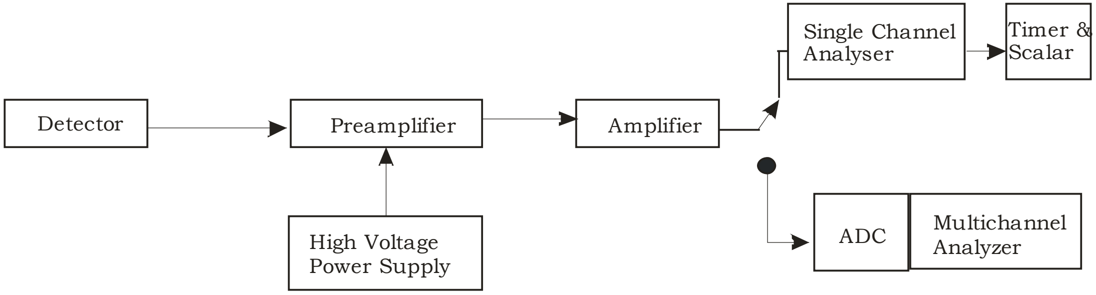


Fig 1.3 Block diagram of an gamma ray spectrometric system

2.1 Classification of Detectors

Some of the detectors which are commonly used for radiation detection and measurement are discussed below:

2.1.1 Gas filled detectors based on the ionization of the gas in a detector

(a) Ion chamber
(b) Proportional counter
(c) Geiger Mueller (GM) Counter

2.1.2 Scintillation detectors based on the conversion of absorbed energy into light

(a) Inorganic Scintillators - NaI(TI), CsI(Na), Bismuth Germanium Oxide (BGO), BaF_2 and ZnS(Ag).
(b) Organic scintillators - Anthracene and stilbene crystals, plastics and some fluorescent chemicals.

2.1.3 Semiconductor detectors based on the collection of charge carriers (electrons/holes) produced in the depleted region of the semiconductor

(a) Lithium drifted germanium (Ge(Li)) and High Purity Ge (HPGe).
(b) Lithium drifted silicon (Si(Li)).
(c) Silicon surface barrier detector.

2.1.4 Neutron detectors based on nuclear reactions and subsequent detection of emitted charged particles

(a) Gas filled detectors with BF_3, ^{3}He and ^{4}He as fill gases
(b) Fissile material coated detectors
(c) Scintillation detectors (loaded plastics)

2.1.5 Miscellaneous Detectors

(a) Solid state nuclear track detectors (SSNTD)

(b) Cherenkov detectors
(c) Photographic emulsions
(d) Thermoluminiscence dosimeters.

The working principles of some of these detectors are described below:

2.2 Gas Filled Detectors

Gas filled detectors are generally used for detection of alpha and beta radiations, and in special cases for γ radiations. A gas filled detector has a central wire kept at a positive potential and a metallic body kept at a negative or zero potential. Methane, ethane, LPG, etc. are some of the gases (fill gases) that are used in these detectors. A schematic diagram of such a detector is shown in Fig. 1.4. For beta particle detection, the gas chamber is sealed with a thin window through which the radiation enters. For alpha particle detection, a special arrangement is made to introduce the alpha source into the detector since the range of alpha particles is very small. When charged particles enter the gas chamber, they produce ionization and/or excitation in the gas. The primary ionization is proportional to the energy absorbed in the gas. Voltage applied across the electrodes provides the necessary electric field for the collection of electrons and ions produced. Usually the primary ionization is small (about 3×10^4 ion pairs/ MeV) and accordingly the signal (current) is also small. To improve the signal, high voltage is applied across the electrodes which results in secondary ionization and the number of ion pairs produced are increased by several orders of magnitude. The variation of current due to the flow of electrons/ions or the resultant voltage across an RC coupling (pulse height), as a function of the applied voltage is shown in Fig.1.5. Regions of importance and multiplication process are described below.

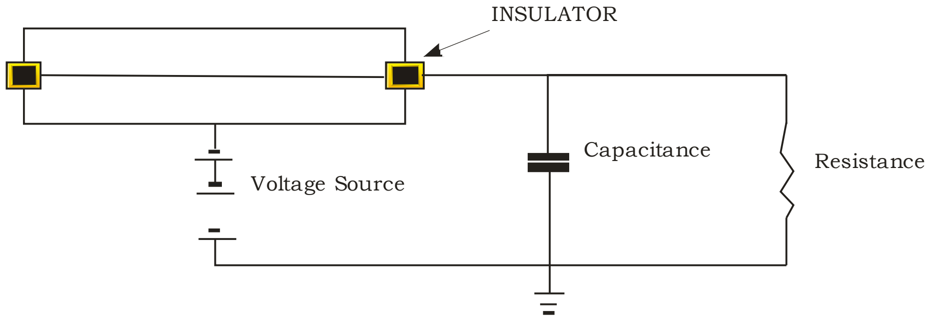


Fig.1.4 Schematic diagram of a gas filled detector for pulse operation

2.2.1 Ionization and Recombination Region (I)

The radiations that enter the detector interact with the fill gas in the detector and produce ionization. Only a small voltage is applied in this region. Under the influence of the applied voltage, electrons and positive ions are attracted towards anode and cathode respectively. As the applied voltage is small, there is a possibility that some of the ion pairs might recombine. Thus competition between ionization and recombination leads to a slow rise of pulse height with the applied voltage.

2.2.2 Saturation Region (II)

In this region of applied voltage (V_0 to V_1), all the ions produced in the primary ionization are collected at the respective electrodes. Thus, pulse height remains constant in this region. This region is known as saturation region. The total number of ions produced, and the resultant pulse height, are proportional to the energy of the charged particle. Detectors operated in this region are called ionization chambers. They are used for the measurement of total radioactivity and radiation spectroscopy as well as for particle detection in heavy ion reactions.

2.2.3 Proportional Region (III)

With further increase in the applied voltage (V_1 to V_2), the electrons, produced in the primary ionization, get accelerated resulting in their enhanced kinetic energy (KE). When the KE of these electrons exceeds the ionization potential of the fill gas, secondary ionization is caused. The probability of secondary ionization increases exponentially with the increase in the applied voltage. This process is called gas multiplication (actually it is multiplication of ionization in the gas) and the multiplication factor (M) is of the order of 10^4 to 10^6. It means that about 2 x 10^5 ions and electrons produced in the primary ionization by a 6.8 MeV α particle, will be multiplied by 10^4 to 10^6 resulting in 2 x 10^9 to 2 x 10^{11} ions and electrons. Gas multiplication is constant at a given voltage and, therefore, the pulse height is proportional to the absorbed radiation energy. Detectors that are operated in this region are called proportional counters. Since the information on both energy and intensity of the radiation can be obtained, proportional counters are very useful detectors to measure α, β and heavy ions. Proportional counters with BF_3 or ^3He as fill gas are used for neutron measurement.

2.2.4 Region of Limited Proportionality (IV)

As the applied voltage is further increased beyond V_2, the secondary ionization continues to increase. Electrons reach anode quickly but positive ions migration towards cathode is slow. It leads to an increased concentration of positive ions around the anode and results in lowering of the electric field. Although gas multiplication in this region is more than 10^6, the pulse height is no longer proportional to the absorbed energy. This region is not useful for radiation detection.

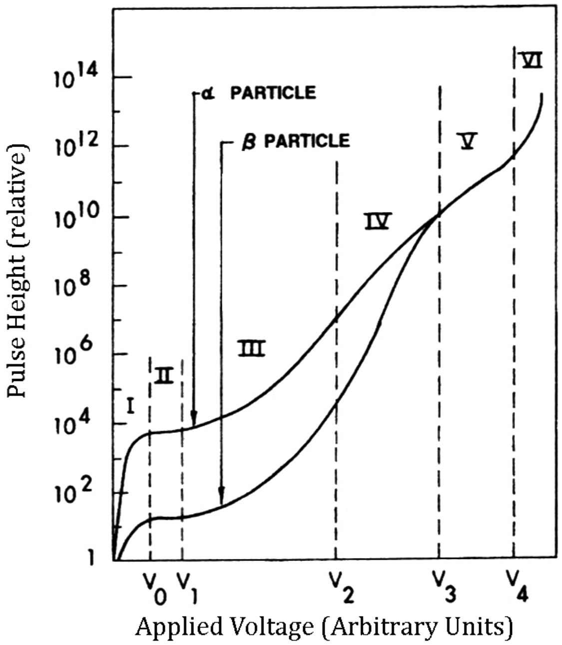


Fig. 1.5 Variation of pulse height (number of ion pairs) with applied voltage in a gas filled detector.

2.2.5 Geiger Mueller (GM) Region (V)

In this region the secondary ionization is so high that positive ions are left uncollected near the anode, thus causing a drop in the effective voltage and termination of gas multiplication. Therefore, irrespective of the number of primary ions produced due to interaction of an event (radiation), the pulse height after multiplication will be same in this region. The pulse height is thus independent of primary ionization. Detectors which operate in this region are called GM counters and are used to measure only the intensity of radiation. Measurement of the energy of radiation is not possible.

2.2.6 Arc or Discharge Region (VI)

Electrons produced in the matter, migrating towards the anode, may excite molecules/atoms of the fill gas during collisions. These excited molecules/atoms deexcite by emitting photons in the visible and UV regions if one of these photons interacts by photoelectric absorption either on cathode surface, or with the constituents of the fill gas; a photoelectron is produced which can migrate towards anode and trigger another avalanche. This effect is negligible in the proportional region (region III, M is 10^4 to 10^6) whereas it becomes significant in the GM region (region V, M $=10^8$-10^{10}). Increase in the applied voltage beyond GM region, would require a large build up of positive charge to terminate the multiplication process. However, this is not effective in terminating the gas ionization since enhanced multiplication is triggered by photoelectric process and not by primary ionization process. Therefore, pulse height begins to increase with voltage leading to discharge of the fill gas. If the detector is operated in this region, then its lifetime is reduced. This region is not useful for detection.

2.3 Geiger-Mueller (GM) Counter

Different detectors have been developed with operating voltages corresponding to regions II, III and V depending on the radiation measurement requirements. Some details regarding Geiger Mueller counter are given below:

The GM counter is one of the oldest radiation detectors. Due to its simplicity, low cost and ease of operation, it is still widely used in many fields. A photograph of GM Counter with accessories is given in Fig. 1.6. Helium and argon are typical fill gases used in GM counters. The counter is operated in the region V and the voltage is around 700 V. As mentioned earlier, all pulses from a GM counter are almost of the same amplitude (~mV) irrespective of the energy of the incident radiation. Therefore, GM counters cannot distinguish different types of radiations. However, the high multiplication factor results in larger pulses, which simplifies the electronic circuitry required for counting and is very advantageous. Large pulses, however, lead to slow rise time of the pulse and hence higher dead time (100-500 μs)[6] of the detector.

Fig. 1.6 Photograph of GM Counting system.

---

[6] Interaction of radiation results in the production of a burst of charge in the detector volume. While this charge is being converted into a voltage pulse, if another burst of charge is produced due to incoming radiation, it will not be processed by the detector as it is busy. Therefore, the detector appears to be dead during this period which is known as the dead time of the detector.

2.3.1 Multiple Pulsing

The positive ions produced due to radiation interaction in a gas filled detector slowly drift away from the anode wire and ultimately arrive at the cathode. They are neutralized by combining with electrons from the cathode surface. In this process, an amount of energy equal to the difference of ionization energy of the gas and the energy required to liberate an electron from the cathode surface is released. If this energy exceeds the cathode work function, it is energetically possible that another electron can be liberated. This electron will then drift towards the anode and may trigger another avalanche, leading to a second full Geiger discharge. The entire cycle will now be repeated. Under these circumstances the GM counter, once initially triggered, would produce a continuous output of pulses. Prevention of multiple pulsing is called quenching. Multiple pulsing is quenched either externally (electronically) or internally (by the use of suitable additives to the fill gas).

2.3.1.1 External quenching

In the external quenching, the operating voltage is lowered to below the threshold of Geiger discharge electronically for a suitable period till all the ions are collected. This will prevent the multiple pulsing.

2.3.1.2 Internal quenching

The fill gas is mixed with a quench gas, which either has (i) a lower ionization potential and a more complex molecular structure than the primary gas or (ii) a gas with higher electron affinity. Typical concentration of the quench gas is in the range of 5 to 10%. Complex molecules prevent multiple pulsing through the mechanism of charge transfer collisions. The primary ions transfer the positive charge to the quench gas molecules. When these charged molecules are neutralized at the cathode, the excess energy goes into the dissociation of these molecules. Examples of quench gases of this type are ethyl alcohol and ethyl formate. An organic quenched tube may typically have a limit of about 10^9 counts as its useful lifetime. Examples of the second type of quench gases are the halogens (chlorine or bromine). Halogen molecules undergo atomic dissociation subsequent to exchanging energy with the positive ions in the fill gas. They dissipate energy non-radiatively and recombine to form halogen molecules. Halogen quenched GM tubes can have infinite lifetime and such tubes are available nowadays.

2.3.2 Types of GM Tubes

Geiger Tubes are useful for β and γ counting. They are available in many forms. The tubes meant for γ counting are enclosed in a metal tube having wall thick enough to stop beta particles. Tubes to be used for β counting are provided with an end window. The window may consist of a thin sheet of aluminum of thickness about 7 mg cm^{-2}. For low energy β counting e.g. βs from ^{14}C (0.15 MeV), thin mica end windows of 2 mg cm^{-2} thickness are used. This type of GM tubes can be used to detect alphas though considerable part of α energy is degraded. It is to be noted that 95-100% of beta particles that enter the GM tube are counted. On the other hand, only a small fraction of gamma rays are counted as a large fraction passes through the tube without interaction.

2.4 Scintillation Detectors

Some substances scintillate, i.e. emit light, on absorption of radiation. The emission process is either fluorescence or phosphorescence. The process of phosphorescence, due to its long decay time, is not desirable in a detection material. The intensity of fluorescent light output is a measure of incident radiation energy. The scintillation detector is coupled to a photomultiplier tube (PMT). Electrons are produced consequent to photoelectric absorption of fluorescent light at the surface of the photocathode of PMT. These electrons are multiplied over many dynodes in the PMT and collected at the anode to produce the primary signal. The signal is processed to get information on the intensity and energy of radiations. Both inorganic as well as organic scintillators are used as detector materials.

Important properties of a good scintillation material are : (i) They should convert the radiation energy into detectable light with high scintillation efficiency, (ii) Light yield should be proportional to the deposited energy, iii) The detector material should be transparent to the wavelength of its own emission, (iv) The decay time of the induced luminescence should be short ($\sim 10^{-9}$ s), (v) It should be possible to make scintillation material into large size detectors of desired shape and (vi) The refractive index should be near to that of glass ($\mu=1.5$) to permit efficient coupling with a photomultiplier tube.

2.4.1 Inorganic Scintillators

Crystals of alkali halides such as NaI and CsI are good inorganic scintillators. Energy gap between the valence and conduction bands in these scintillators, is of the order of 5-6 eV. When the radiation energy is absorbed, electrons from the valence band are excited to the conduction band leaving holes in the valence band. Electrons and holes move in the detector volume. De-excitation is accompanied by the emission of photons. The wavelength of emitted photons is in the UV region. Since most of the photomultiplier tubes have good sensitivity for photons of the visible region, wavelength of the photons has to be shifted to visible region. This is achieved by doping CsI/NaI crystals with impurities (e.g. 1 % Tl) that create intermediate donor and acceptor sites in the band gap. These dopants are called activators. In NaI((Tl) crystal about 30 eV of energy is needed to produce a photon in the visible region. On an average 10 photons are needed to liberate one photoelectron by photoelectric absorption at the photocathode surface of PMT. Thus on an average about 100-300 eV of energy is required to liberate an electron at the photocathode surface. This limits energy- resolution of the detector to 7-10%. Despite the poor resolution, scintillation detectors are widely used due to their high detection efficiency (25 to 100%). They are available in desired sizes and shapes. Other scintillators like CsI(Tl), CsI(Na), LiI(Eu), BaF_2 and BGO are also used in the radiation measurement.

2.4.2 Organic Scintillators

Mixtures of certain organic chemicals serve as good liquid scintillators. One such mixture consists of a solvent such as dioxan in which a scintillator 2,5-diphenyl oxazole (PPO) is dissolved. The radiation energy is absorbed by the solvent molecules and then transferred to solute PPO, which gives fluorescence. As the emitted light is in the ultraviolet region, a small amount of 1,4 bis-[2-(5-phenyloxazolyl)]-benzene (POPOP) is added to shift the wavelength to the visible region.

In organic scintillators, absorption of radiation energy results in excitation of the molecules from their electronic ground state (S_0) to higher states such as S_1, S_2 and S_3. The excited states, which have very short life time (10^{-9} s), de-excite to the ground state via fluorescence. Triplet states have a longer life time (> ms) and de-excitation through these states is called phosphorescence. This component is not suitable for detection due to slow decay characteristics.

Liquid scintillators are very useful where large volume detectors are required. They are also used for alpha and beta counting with nearly 100% efficiency. Radioactive sample is added to the liquid scintillator in a suitable vial. This vial is kept in a light reflector that is coupled to a PM tube. Interaction of radiation with the scintillator is nearly 100%, leading to a high detection efficiency.

Plastic scintillators, which are solid solutions of organic scintillators, have fast response time and are useful in nuclear spectroscopy. Most widely used plastics are polystyrene, polyvinyl toluene and polyphenyl benzene.

2.5 Semiconductor Detectors

In a semiconductor, the band gap between the conduction and valence bands is of the order of 1 eV. Semiconductors can be of n-type (electron rich) or p-type (electron deficient) depending upon the type of impurities present in the crystal. A semiconductor detector is a diode with p-n junction. Application of a reverse bias across the diode results in the transport of electrons towards the positive electrode (n-end) and that of holes towards the negative electrode (p-end), thus creating an intrinsic region with no charge carriers. This charge carrier free region is the active volume of the detector. The incident radiation interacts with the detector material and, produces electrons and holes in the active region. Under the influence of applied field the electrons and holes are collected at the respective electrodes to give a voltage pulse which is proportional to the energy of the incident radiation. On an average about 3 eV of energy is required to produce an electron-hole pair. Thus a large number of electron-hole pairs are formed compared to those in other types of detectors. This results in very good resolution. The small band gap leads to some leakage current due to thermal population in the conduction band at room temperature. Leakage current is reduced by cooling the detector to liquid nitrogen temperature. Lithium drifted Ge detectors, (Ge(Li)) has been a high resolution detector. However with technology improvement, currently high purity Ge detectors (HPGe) are most widely used in gamma ray spectrometry. The resolution of these detectors is in the range of 1.5 to 2.2 keV, at 1332 keV gamma ray of ^{60}Co, depending on the quality and volume of the detector. The efficiency of the detector is quoted with respect to 3" x 3" NaI(TI) detector. Detectors with relative efficiency of the order of 5 to 20% are commonly available and more recently detectors with 80-100% relative efficiency have also become available.

Silicon surface barrier detectors are most commonly used for spectroscopic measurement of alpha particles and fission fragments. These detectors are thin (in the region of 100 microns to a few mm) and have resolution of 15 to 20 keV at 5 MeV alpha particle energy. Si(Li) detectors find wide applications in conversion electron and X-ray spectrometry, and in X-ray fluorescence (XRF) measurements. The resolution of Si(Li) detectors is of the order of 150 to 250 eV at 15 keV. Other types of detectors are diffused junction, ion implanted and totally depleted detectors.

2.6 Miscellaneous Detectors

2.6.1 Cherenkov Detectors

If the velocity of a charged particle in a medium (phase velocity) is greater than the speed of light (c) in that medium, then light is emitted when the charge particle passes through the medium. This radiation is known as Cherenkov radiation. This effect is observed only if the velocity of the radiation is comparable to the velocity of light (relativistic region). Electrons with energy of 265 keV have a velocity of 0.751 c. They can be detected if they are passing through water (refractive index, p = 1.332). Velocity of β-rays is not changed whereas phase velocity of light is c/μ = c/1.332 = 0.750 c. Therefore, Cerenkov radiation is emitted. This light can be utilized to detect β^- rays of high energy.

2.6.2 Photographic Emulsions

Ordinary photographic films consist of an emulsion of silver halide grains suspended in a gelatin matrix and supported on a glass or cellulose acetate film. Ionizing radiation sensitizes silver halide grains and sensitization remains intact for long periods. Generally after a periodical period, the film is developed. Extent of sensitization is proportional to the cumulative exposure to radiation. Examples of photographic emulsions are radiographic films and nuclear emulsions. These detectors are very useful in health physics applications, nuclear spectroscopy and radiography.

2.6.3 Thermoluminescence Dosimeters

In some of the inorganic crystals, electron hole pairs formed due to radiation interaction are trapped in the crystal. On heating, these defects combine and the energy released leads to the emission of light. The intensity of the emitted light is proportional to the absorbed energy. These materials are used for dose monitoring and the detectors are known as thermoluminiscent dosimeters (TLD). e.g. $CaSO_4$:Dy, LiF and CaF_2:Mn. TLDs are used to measure the cumulative dose to individuals working in a radioactive laboratory.

2.6.4 Track-etch Detectors

Ionizing radiations like α particles and fission products, having high linear energy transfer (LET) in a medium, while passing through a dielectric material create a trail of damaged molecules along their path. In some materials, the tracks can be made visible upon etching in a strong acid or alkali solution. The damaged molecular tracks are etched faster than the bulk and look like pits on the surface. Number of tracks are related to the intensity of interacting radiation and, therefore, are very useful for measurement of radiation. These tracks can be counted by using a microscope. As each impinging ion leaves a track in the detector, detection efficiency is 100% making it very useful for low level radiation measurements. Commonly used track detector materials are quartz, mica, silica glass, flint glass, polyethylene terphthalate, lexan, makrofol, cellulose triacetate, and cellulose nitrate. These detectors are useful in the nuclear reaction studies and radioactivity measurements.

Chapter 2

FUNDAMENTALS OF RADIOCHEMICAL PRACTICE

Radioactive substances emit ionizing radiation and are, therefore, handled in specifically designed laboratories. The difference between a conventional chemical laboratory and a radiochemical laboratory depends upon the amount of radioactive substance handled and the nature of chemical operations. This chapter outlines the different classes of radiochemical laboratories, radiochemical practices and commonly used radiochemical separation procedures. Most of the experiments described in this book involve handling of small amounts of radioactivity and can be carried out in a suitably altered conventional chemical laboratory.

1. RADIOCHEMICAL LABORATORIES

1.1 Introduction

Radiochemical laboratories are designed to ensure that there is no spillage or release of radioactive materials outside the designated area in the laboratory. Accordingly, these laboratories have features to ensure that any radioactive substance, which may get airborne during the handling of open radioactive sources, does not enter the laboratory air. Open sources of radioactivity are, therefore, always handled in fume hoods or glove boxes, which have suitable ventilation arrangements to operate at a slightly negative pressure with respect to ambience to prevent the release of radioactivity into the laboratory air. The surfaces where radiochemical work is carried out, are chosen such that they can readily be cleaned in case of an inadvertent spill of the radioactive material. Therefore, surfaces in the fume hoods/glove boxes and table tops are covered with polyethylene sheet and laboratory floor is covered with polyvinyl chloride (PVC) tiles to help in rapid cleanup in case of a spill. These precautions help prevent contamination of air, surfaces or persons with radioactive materials. The type of ventilation arrangement and flooring of the laboratory depends upon the level of radioactivity to be handled.

1.1.1 Classification of Laboratories

Depending on the type and the quantity of the radioactive material to be handled, radiochemical laboratories are classified into three categories as given in Table 2.1. Additionally, depending upon the type of operation involved, the quantities of radioactivity handled are modified by the factors shown in Table 2.2. These modifying factors apply to C and B type laboratories. For example, although a maximum of 10 µCi of radioactivity is allowed in a type C laboratory, sealed samples up to 1 mCi can be handled if only storage is envisaged as the modifying factor is 100. In the case of dry and dusty operations, radioactive sources having 10 µCi can not be handled in type C laboratory, as the modifying factor is 0.01. These operations are carried out only in a type B laboratory.

Table 2.1 - Quantities of radioactivity handled in different types of radioactive laboratories.

Type of Laboratory	Quantity of Radioactivity handled (Open source)
Type C	10 µCi (3.7 x 10^5 Bq) or less
Type B	10 µCi to 10 mCi
Type A	Above 10 mCi

Table 2.2 - Modifying factors for different operations to calculate the quantity of radioactivity handled.

Sl. No.	Operation	Modifying factor
1.	Storage only	x 100
2.	Very simple wet operations (pipetting, making up etc.)	x 10
3.	Normal chemical operations (precipitation, solvent extraction)	x 1
4.	Complex chemical operations with risk of spillage	x 0.1
5.	Simple dry operations (planchetting, drying precipitate etc.)	x 0.1
6.	Dry and dusty operations	x 0.01

1.2 Specifications for Different Types of Radiochemical Laboratories

1.2.1 Type C Laboratory

Type C laboratory is meant for carrying out practical experiments recommended at university laboratories and similar research institutions. In Type C laboratories, β, γ radioactive materials in solution form as well as sealed sources are handled. Among β-γ radioactive materials, isotopes like: ^{3}H (12.32 y), ^{14}C (5730 y), ^{32}P (14.3 d), ^{35}S (87.5 d), ^{60}Co (5.27 y), ^{131}I (8.02 d), ^{137}Cs (30.17 y), ^{140}Ba (12.75.d) and ^{144}Ce (284.8 d) are allowed to handle in C type lab. Small amounts of alpha emitters such as natural uranium and thorium or products in the natural decay series can also be handled in these laboratories. Maximum quantity of radioactivity in the solution form used for each experiment shall not be more than 10 μCi. A clean, well-equipped chemical laboratory having one or more fume hoods will qualify for designation as a type C laboratory. The fume hood should have an exhaust fan, providing a linear flow of air across the front opening (at 30 cm opening) at a velocity not less than 30 m per minute. Photograph of a typical fume hood is shown in Fig. 2.1. There is no need for filtration of air from this laboratory, before discharging it into the atmosphere.

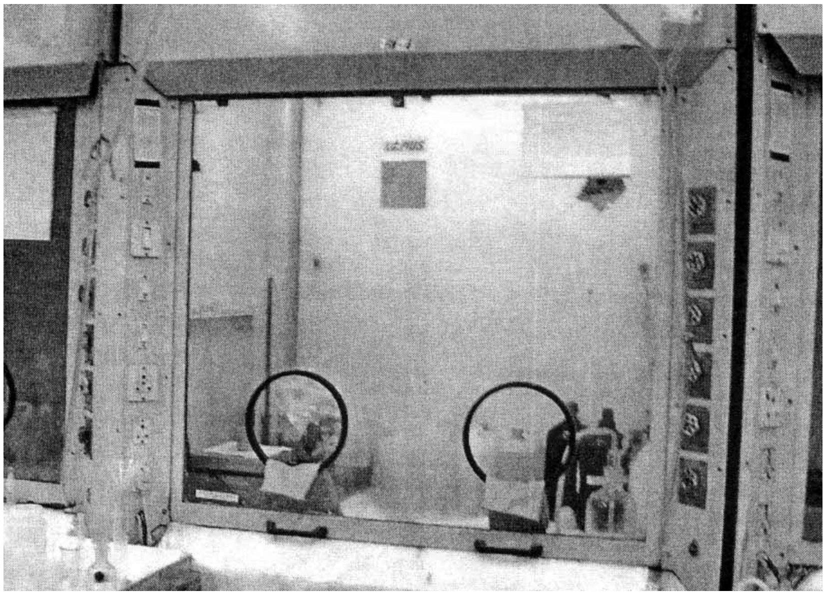


Fig. 2.1 View of a typical fume hood used in radiochemical operations.

Steel or wooden work benches and cupboards are allowed in this type of laboratory. But the top surface, which is used for laboratory work, shall be covered with non-absorbing plastic laminates, which are easy to decontaminate in the event of a spill. Each laboratory shall have a sink/wash basin made of stainless steel, or porcelain. These sinks should have taps with handles, which are elbow operated. Effluent from these laboratories can be mixed with general effluents. Fume hoods should be kept close to the walls, adjoining an open space, so that the exhaust duct can be taken out through a hole in the wall and discharged to the open atmosphere. A separate room for housing the counting equipments is preferable. Otherwise, a portion of the laboratory can be partitioned and converted into a counting room.

1.2.2 Type B Laboratory

Type B laboratories have all the specifications of type C laboratories and the following additional features. Any type C laboratory can be upgraded to type B with a few additions. As larger quantities of radioactivity is handled, it is essential to display "radiation symbol" at the entrance of the laboratory and also the laboratory shall have "restricted entry". A physical barrier separates this laboratory from other areas. Persons working in the laboratory are required to wear shoe covers before entering the laboratory and remove them before leaving the laboratory. This precaution limits the contamination to the laboratory in the event of an inadvertent spill. Wash basins and a contamination monitor are installed at the entrance, and personnel must wash their hands and monitor themselves for radioactive contamination before going to other areas. Since the quantity of radioactivity is more, lead bricks for making shielded enclosures in fume hoods may be necessary. Fume hoods should be strong to bear the weight of the lead shielded enclosures. Laboratory exhaust is through fume hood only, and High Efficiency Particulate Air (HEPA) filters are to be used in the exhaust line to ensure all particulate contaminants are retained before discharging the air into the atmosphere. The exhaust air is continuously monitored for any radioactive contamination. Liquid waste containing radioactivity should be collected separately, monitored and discharged only after proper dilution. Solid wastes have to be monitored before disposal. If contaminated with long-lived radioisotopes, the waste has to be disposed of by the recommended procedures.

1.2.3 Type A Laboratory

Type A laboratories include laboratories for isotope production and laboratories handling alpha emitters in large quantities. Activity more than 10 mCi should be handled only in a type A laboratory. Well laid out zones (of section 1.2.4), barriers and fool-proof ventilation systems, with standby provision for each system, are essential. Supply air is also filtered and cooled. Each zone has continuous air monitors and background dose monitors with audible alarms if the dose level exceeds the preset values. Separate exhaust systems for fume hoods and glove boxes are desirable. Elaborate safety systems for fire indication and fire fighting are provided. Low level alpha radioactivity is handled in the fume hoods. Chemical operations involving high level alpha radioactive materials are carried out in leak tight glove boxes[1]. Photograph of a typical glove box is shown in Fig. 2.2. Shielded cells with remote handling facility and lead glass viewing systems are required to handle the

---

[1] Glove box is a metal/perspex box enclosure which is connected to an exhaust system. It is always maintained under slightly negative pressure. All the operations are carried out using rubber gauntlets fixed at different heights on viewing panel. Materials in/out operations are carried out using a double door system and a bagging out facility.

material whose (β,γ) radioactivity is in the order of Curies and above. Flooring of the laboratory should be covered with PVC tiles as it is easy to take swipes from the PVC floor for routine monitoring of possible contamination and, to remove and replace them in case of contamination due to spillage. Separate storage tanks for low level waste and medium level waste solutions are provided. Washings from laboratory are not discharged to public drains. Solid wastes have to be packed, monitored and sent for disposal to specially designated disposal sites.

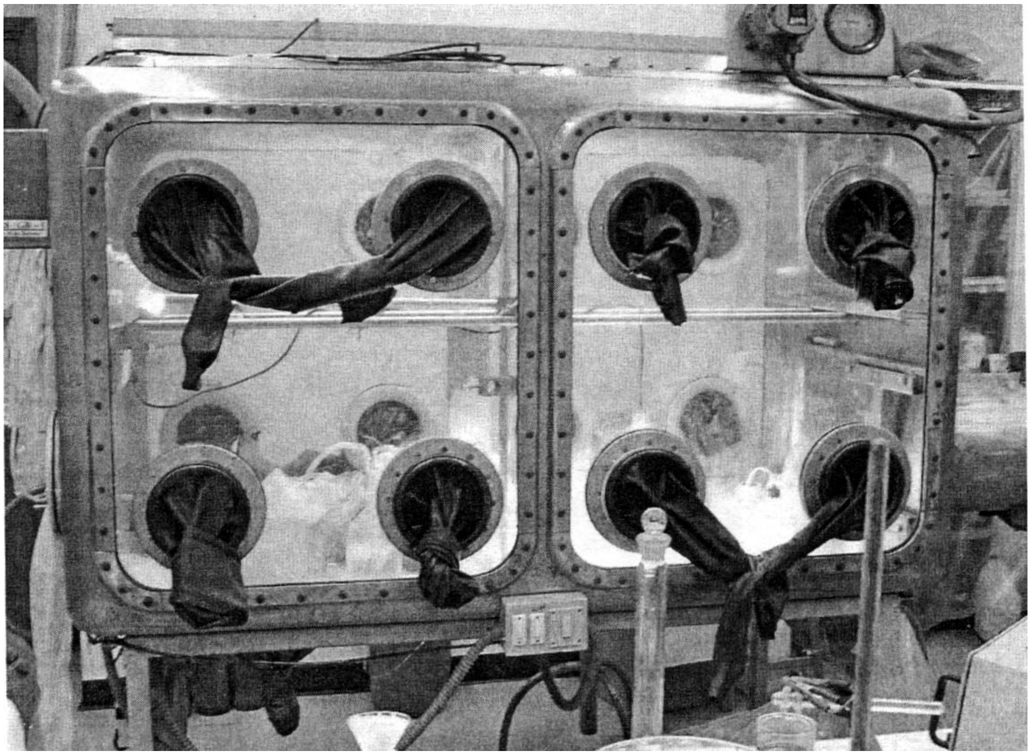


Fig. 2.2 Photograph of a typical glove box used in type A laboratory.

1.2.4 Classification of Radioactive Zones in a Radiochemical Laboratory

A Radiochemical laboratory, particularly of class A type, is divided into four different zones.

(a) White Zone

In this area no radioactive materials are handled. Office rooms, reception room, workshops etc. constitute the white zone. Probability of contamination is nil.

None of the following materials is permitted beyond the white zone:

1. Eatables and beverages,
2. Smoking items and tobacco,
3. Chewing materials and,
4. Handbags containing lipsticks, cosmetics or articles of similar nature.

(b) Green Zone

In this area no radioactivity is present but possibility of contamination is not ruled out, because of its proximity to areas handling radioactivity (amber zone). Protective clothes are not required. This zone houses cabins for personnel working in the laboratories. Health physicist's room, counting room, corridors etc. form part of this zone. Direct fountain (non storage type) water coolers for drinking water may be permitted in the white and green zones.

(c) Amber Zone

Laboratory areas where equipment like fume hoods and glove boxes containing active materials are kept, belong to the amber zone. Possibility of floor contamination is finite and more than in the green zone. Entry into this area is restricted to actual laboratory personnel and that too with protective clothing. A physical barrier separates green and amber zones.

(d) Red Zone

Interior of fume hoods, glove boxes and shielded cells form the red zone. Radioactive materials are handled in this zone. This is a contaminated area and personnel entry is forbidden.

1.2.5 Ventilation

A radiochemical laboratory must have provision for controlled ventilation. In a simple type C laboratory the fume hood, where radioactivity is handled, should have proper exhaust fan such that air flows from the laboratory towards the fume hood at an adequate speed to prevent any radioactive aerosol from contaminating laboratory air. In B and A type laboratories, the flow of air is unidirectional, from white zone to green, to amber and to red zones. This flow is guided by keeping the fume hoods and glove boxes at a negative pressure by providing suitable exhaust fans. The exhaust air is passed through HEPA filters to remove any particles which may have radioactivity associated with them. The design of ventilation provides the gradual increase of negative pressure from white to green, to amber to red zones.

1.3 Precautions to be observed in a Radiochemical Laboratory

The laboratory techniques in radiochemical methods call for certain bench practices while dealing with radioactive isotopes. The foremost among them is the safety aspects to be followed in a radiochemical laboratory. The rules and regulations essentially define measures meant to prevent the release or spread of radioactivity, which may contaminate air, work place and personnel. To ensure accountability, one of the persons in the active laboratories is designated as laboratory-in-charge and his/her name is prominently displayed at the entrance.

Some of the rules to be followed by the working personnel in the laboratory of type C are as follows:

1. All work with radioactive materials should be carried out preferably in a fume hood. Polythene sheet must be spread in the fume hood before starting an experiment to help in rapid clean up.
2. Eating, drinking, smoking and using snuff are prohibited in the laboratory.
3. While working with radioactive materials, personnel should wear surgical gloves to avoid direct physical contact with the radioactive substance.
4. No work with radioactive material should be carried out by anybody having an open cut, skin lesion or injury.
5. Pipetting of any solution should not be done by mouth in a radioactive laboratory. Pro-pipette or a syringe should be used.
6. Any spillage of radioactive solution, contamination of personnel or work area or any accident must be reported immediately to the lab- in-charge.

7. All radioactive materials must be sealed and labeled properly, with date and responsible person's name written on it.
8. Radioactive samples should not be removed from the laboratory without the permission of lab-in-charge.
9. After removing the gloves, hands should be washed with detergent solution and water.

Rules are further stringent in the type B and A laboratories as the level of radioactivity handled is much higher. Wet, dry and dusty operations are permitted in these laboratories. Therefore, necessary care regarding ventilation, protective clothing, waste disposal etc. has to be taken. All the rules described for type C laboratory are applicable to type B and A laboratories. Additionally following rules have to be followed.

10. A health physicist should oversee all operations.
11. As described in section 1.2.4, the radioactive area (amber zone) is separated from the green zone by a barrier. Persons entering the amber zone should wear the protective clothing like shoe covers and laboratory coat.
12. All the work should be carried out in fume hoods or glove boxes. Polythene sheet must be spread in the fume hoods/glove box, before starting work. It is advisable to spread a polythene sheet on the floor, in front of the glove box/fume hood, while working. This should be removed and disposed off after work and the floor monitored for contamination.
13. A film badge or a Thermo Luminescent Dosimeter (TLD) for personnel monitoring of radiation exposure must be worn by the person while working in the radioactive laboratory.
14. All the operations with exposed sources of radioactivity must be carried out in a fume-hood with proper ventilation.
15. Liquid and solid wastes must be separated and stored in separate containers.
16. Hands should be washed and monitored using hand monitors provided at the barrier in the green zone of the radioactive laboratory. Only when the hands are free from contamination, personnel should leave the laboratory.

1.4 Introduction to Radiochemical and Radioanalytical Methods

Chemical or analytical procedures involving the use of radiation (mainly from radioisotopes) are termed as radiochemical or radioanalytical methods. Some of the commonly used methods are given below.

1.4.1 Precipitation

The amounts of radioactive material to be isolated is sometimes very small and some operations like normal precipitation are not feasible. For example 10^6 Bq of ^{134}I, with a half-life of 52.6 m, corresponds to 4.55×10^9 atoms, which is 1.01×10^{-12}g. Thus the amount of substance to be isolated in a radiochemical separation is very low. This poses problems for conventional separations such as precipitation and filtration. Material might be lost on the surface of glassware and filter paper. To overcome this problem, the technique of co-precipitation is used. In this technique stable isotope(s) of the element of interest, in the form of a soluble salt, is added in milligram quantities to the solution containing the radioisotope before carrying out precipitation. The oxidation state of the element in the salt should be same as that expected for the radioisotope, or a suitable valence adjustment procedure should be used. On precipitation, weighable quantity of precipitate is thus obtained and can also be used to determine the yield of precipitation process. Counting the precipitate gives the quantity of radioisotope present in the starting solution. For example, in the fission of ^{235}U, ^{140}Ba and some other isotopes of barium are formed. Traces of barium produced in the fission can be isolated by adding inactive barium (10-20 mg) in the form of barium nitrate solution and precipitating it as $BaCrO_4$. In some cases when isotopic carrier is not available, salt solution of an element having similar chemical properties is added. For example, radium can be co-precipitated with $BaSO_4$. It is possible that other radioactive elements (isotopes) present in trace amounts may be adsorbed on the precipitate. This can be avoided by using a carrier, which acts as a hold back carrier for the unwanted material. For example, while precipitating radioiodine using inactive iodine carrier, radio-tellurium might be adsorbed on the AgI precipitate. This is prevented by adding Te carrier to the solution prior to precipitation of AgI. Inactive Te carrier holds back most of the radioactive Te from being adsorbed.

While isolating a radioelement from a host of other radioactive elements, as in the case of fission products, it is possible that many radioelements are adsorbed on the precipitate. The element of interest is separated and purified from unwanted elements by repeated dissolution and precipitation cycles. Unwanted elements can also be scavenged using a carrier like Fe^{3+} and precipitating $Fe(OH)_3$. The $Fe(OH)_3$ precipitate has the ability to occlude many ions that do not have carriers. Fe^{3+} carrier acts as a scavenger for most of the unwanted radioelements. Carriers, hold back carriers and scavengers are used in the separation and purification of strontium from fission products and details are described in the experiment 17 in Chapter 5.

1.4.2 Solvent Extraction

Solvent extraction is often employed for the separation of radioisotopes. It is based on the difference in the distribution of the solute (metal ion) in two different phases, usually aqueous and organic phases. Normally metal ion of interest is extracted from the aqueous phase into the organic phase, leaving behind other unwanted ions in the aqueous phase. The metal ion in the organic phase can then be back extracted into a suitable aqueous medium for further use. This technique can effectively be used to separate an element of interest from a host of other elements. For example, in fuel reprocessing uranium and plutonium are separated from a large number of fission product elements by extraction into tri-butyl phosphate (TBP). Thorium-234 can similarly be separated from natural uranium by selective extraction of uranium by amines from a chloride medium.

1.4.3 Ion Exchange

Ion exchange is another versatile separation technique in which anions or cations are exchanged on a column consisting of suitable exchanger materials e.g. natural or synthetic exchangers such as zeolites, Dowex-1 and Dowex-50. Apart from separations, ion exchangers are useful for concentrating the substances. Since limited number of elements form anionic species in a given medium, anion exchange usually provides better separation from a large number of elements when the ion of interest is held on the column as anionic species. For example, in 7-8 M nitric acid tetravalent Np or Pu are present as anionic species and can be held on an anion exchange resin for separation from many other elements. Cation exchange is usually employed when elements with similar chemical properties have to be separated. Separation is usually achieved during elution using complexing agents. On the other hand, when the chemical properties of the elements are widely different, a proper pH control during either loading or elution also helps in achieving a good separation.

1.5 Preparation of Samples for Counting

Radioactivity in a solid/liquid sample is measured by counting appropriately prepared samples. In the case of alpha counting of solid samples particular care has to be taken to make thin sources of the radioisotope samples. As alpha particles have short ranges, they might be attenuated if thick samples are made. Sample to detector geometry must be reproducible. For example, source to detector distance, or volume of a liquid sample in a well type counter, should be fixed. Labelling the samples by writing the name of the isotope, date and user's name is not only a good practice but essential in radiochemical work.

1.5.1 α-counting

A small quantity of solution (50 μL) containing α-emitting radioisotope is transferred on to a thin metallic disc, known as planchette. It is dried slowly under an infrared lamp. The planchette is carefully heated to red heat and cooled for improving adherence of radioactivity to the metal surface. Sources prepared thus are called point sources or weight less sources. However, for energy measurements, ideally, mono-layer samples are needed. For this, radioisotopes are electrodeposited on either polished stainless steel or platinum discs.

In the case of liquid scintillation counting, samples are prepared by directly transferring radioactivity in solution form to scintillator solution (5 mL) kept in a counting vial. Volume of the scintillator is decided by the required sample to detector geometry. Care should be taken to avoid the presence of components in the sample that absorb emitted scintillations. α-samples have to be inserted into the detector in the case of proportional counters and into the vacuum chamber in the case of solid-state detectors like silicon surface barrier detector.

1.5.2 β-counting

As β-particles have a reasonable range, counting samples are prepared in a variety of forms such as point sources and precipitates. These sources are then covered with adhesive tape to avoid direct contact. Samples for liquid scintillation counting are prepared by transferring a known amount of solution into the scintillator solution in a counting vial.

1.5.3 γ-counting

As in β counting, sources for γ-counting are prepared as point sources, precipitate or direct liquid samples. Liquid sample aliquots are taken in special test tubes called counting tubes or counting vials.

β-γ samples are usually measured by keeping them outside the detectors.

Chapter 3

EXPERIMENTS ON RADIATION MEASUREMENT

1. BETA AND GAMMA COUNTING USING A GM COUNTER

GM counter is a frequently used counter for radiation measurement. Photograph of a GM counter with accessories is shown in Fig. 1.6. The GM counter is a sealed cylindrical tube filled with a gas and provided with a thin window for allowing the entry of radiation into the detector volume. Radiation causes ionization of the gas and a positive voltage applied to central wire electrode amplifies the primary ionization. As electrons are collected at the anode, an RC coupling circuit generates an output pulse having voltage in the range of a few mV. This pulse is amplified using a suitable amplifier whose output is connected to a timer-scalar. The pulses received during a preset time interval are recorded. The voltage of the pulse is referred to as pulse height.

Along with electrons, an equivalent number of positive ions are produced in the GM tube. Most of these are produced very close to the anode and take a long time to drift towards the cathode. This results in space charge, which reduces the electric field, for a certain duration (T). If another radiation enters the GM tube during this period, the ionization caused will be lower and will not give rise to a pulse of required height for its registration. Thus the GM tube is not active for a duration T after every event and this time is known as dead time. Due to this, the recorded count rate is less than the actual count rate and extent of loss depends on the dead time. It is, therefore, essential to know the dead time of the system so as to have accurate determination of the source strength. The dead time T of GM tubes is in the range of 100-500 microseconds. The relation between observed count rate (m) and the true count rate (n) is given by:

$$n = \frac{m}{(1 - mT)} \tag{3.1}$$

GM counters are operated in a particular voltage region, 500-800 V. Operating voltage, V, of a GM detector is a constant, which depends on the configuration of the detector. The value of V is determined experimentally by identifying the voltage region in which the observed count rate is nearly constant for a given source strength. This voltage region is called the plateau of that GM counter. Experiment 1 describes the determination of plateau region. In experiment 2, determination of the dead time of the counter is described, while experiment 3 deals with statistical aspects of radiation measurement.

Experiment 1

PLATEAU OF A GM COUNTER

Discussion

In a counting set-up, only pulses having voltage (pulse height) above a certain threshold value are counted, to minimize contribution by the background noise. The process of selecting this threshold voltage is known as pulse height discrimination (PHD). While counting a radioactive source, the size of the pulses in a GM counter essentially depends on the voltage applied to the anode (EHT). At low EHT value (300 V), pulse height is smaller than the PHD and, therefore, these pulses are not counted. As the EHT is increased the pulse height increases and for some pulses it exceeds the PHD value. These pulses are, therefore, recorded. With increase in EHT, the number of pulses having pulse height more than PHD value increases. When all the pulses attain sufficient height, the count rate remains essentially constant with EHT. This region of nearly constant count rate is called the plateau. A typical plateau is shown in Fig. 3.1 where V_S and V_E are voltages at the beginning and the end of the plateau region. GM Counters have a plateau region spanning 100-200 volts with a slope of 1-5%. It is a practice to apply a positive voltage slightly lower than the mid point of the plateau region. Higher values of EHT can lead to the discharge of the fill gas.

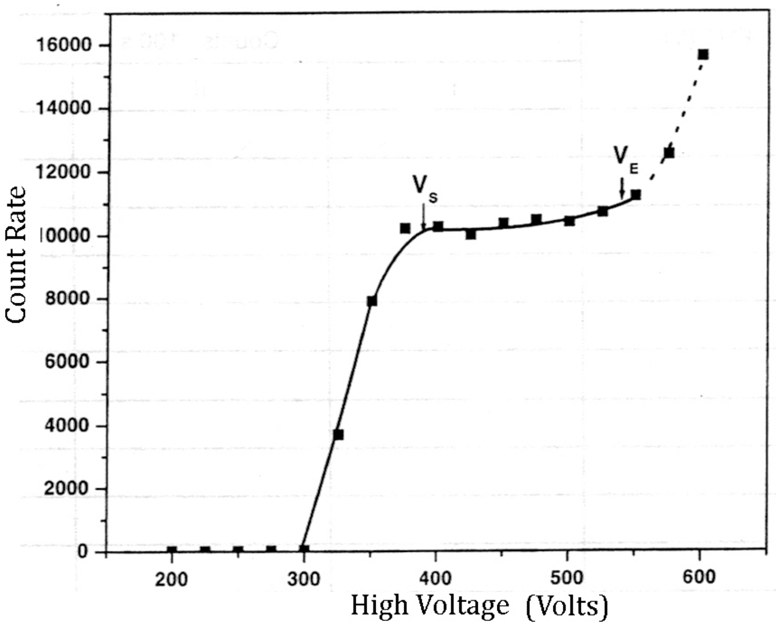


Fig 3.1 Plot of variation of count rate as a function of high voltage applied to a GM counter. Region from V_s to V_e is the plateau.

Materials Required

GM counter setup,β-γ sources :^{137}Cs and ^{144}Ce, and mounting plates.

Procedure

1. Assemble the electronic units of GM counter set up and make connections as given in the instruction manual. Note down the settings.

2. Switch on the mains supply. After a few minutes put on the 'EHT' and wait for 30 min.
3. Mount the given source under the counter window in a shelf of the source holder.
4. Apply a voltage of 300 V and count the sample for 100 s. Note the counts and repeat counting.
5. Increase voltage by 25 V and again take two readings for 100 s each. Record the readings.
6. Continue step 5 until the count rate shows a rapid increase after an initial plateau.

Observations

Date :

GM Counter No.	:		GM tube No.	:	
Function	:	Time	Preset time	:	100 s
Paralysis time	:	250 μs	Operating voltage	:	V
Position	:	Groove No.	Sources	:	^{144}Ce/^{137}Cs

Table 3.1 - Counting data for plateau determination

Sl. No.	EHT (Volts)	Counts/ 100 s		
		I	II	Average C_s
1				
2				
3				
4				
5				
6				
7				
8				
9				
10				
11				
12				
13				
14				
15				

Calculations

1. On a linear graph paper plot count rate (Y-axis) vs. applied voltage (V) (X-axis).
2. Determine the plateau from the graph i.e. the region where the count rate does not change appreciably with applied voltage. From (V_S)_______to V_E_______Volts. Do not increase Voltage beyond V_E. (The experimental plot should resemble Fig. 3.1).
3. Calculate the operating voltage using eqn. (3.2)

$$\text{Operating voltage} = \frac{V_s + V_E}{2} \tag{3.2}$$

4. Determine the slope (S) of the plateau using count rates for V_s and V_E

$$\%S = \frac{C_{v_E} - C_{v_S}}{V_E - V_S} \times 100 \tag{3.3}$$

Experiment 2

DETERMINATION OF DEAD TIME OF A GM COUNTER

Discussion

The dead time (T) of the counter is determined by the method of paired sources. This method is based on observing the count rates from two sources individually and in combination. Because the counting losses are non-linear, the observed count rate due to the combined sources will be less than the sum of the count rates of the two sources counted individually and the dead time can be calculated as follows:

Let n_1, n_2 and n_{12} be the true count rates (samples plus background) with source 1, source 2 and sources 1 and 2 together, respectively. Let m_1, m_2 and m_{12} represent the corresponding observed count rates. Let n_b and m_b be the true and measured background rates.

$$n_{12}-n_b=(n_1-n_b)+(n_2-n_b) \tag{3.4}$$

$$n_{12}+n_b=n_1+n_2 \tag{3.5}$$

The corrected count rate n is given by eqn. (3.1) as $n = m / (1 - mT)$ where m is the observed rate.

Substituting the value of n in eqn. (3.5), one obtains eqn 3.6

$$\frac{m_{12}}{(1-m_{12}T)}+\frac{m_b}{(1-m_bT)}=\frac{m_1}{(1-m_1T)}+\frac{m_2}{(1-m_2T)} \tag{3.6}$$

Solving this equation, a relation for T is obtained in terms of observed count rates as :

$$T=\frac{m_1+m_2-m_{12}-m_b}{2(m_1-m_b)(m_2-m_b)} \tag{3.7}$$

Materials Required

Two sources on aluminium semicircular discs, two dummy aluminium semicircular discs without activity, GM counter with accessories, sample holder and sample stand.

Procedure

1. Assemble the electronic units of GM counter set up and make connections as given in the instruction manual. Note down the settings.
2. Switch on the mains supply. After 10 minutes put on the ‘EHT’. Set the EHT value to the operating voltage obtained in Experiment 1. Allow to stabilize for half an hour.
3. Place the two dummie sources 01 and 02 in the source holder and mount this source holder on a suitable groove of the sample stand. Measure the background count rate m_b. Make duplicate measurements for all the combinations. Carry out all measurements using the same groove of the sample stand.
4. Replace one dummy with source 1 and measure the count rate, m_1.
5. Replace the other dummy with source 2 of the same set and measure the count rate, m_{12}.
6. Replace source 1 with the first dummy and measure the count rate of source 2, m_2.

7. Calculate T using the eqn. (3.7).

Observation

Date	:				
GM Counter No.	:		GM tube No.	:	
Function	:	Time	Preset time	:	60 s
Paralysis time	:	250 µs	Operating voltage	:	V
Position	:	Groove No.	Sources	:	^{144}Ce/^{137}Cs

Table 3.2 -Counting data for dead time determination

Combination		Counts/60 s	Average Counts/s
Source M_1 + Dummy D_2, (m_1)	(i)		
	(ii)		
Source M_1 + Source M_2, (m_{12})	(i)		
	(ii)		
Dummy D_1 + Source M_2 (m_2)	(i)		
	(ii)		
Dummy D_1 + Dummy D_2, (M_b)	(i)		
	(ii)		

NOTE: Count Dummy D_1 + Dummy D_2 for longer duration to get a statistically reliable value of m_b.

Calculations

1. Substitute average values from the above Table in the eqn. (3.7) and calculate T.
2. Calculate true count rates of the sources 01 and 02 using T obtained in this experiment. e.g. When T =100 µs and m =1000 cps, then by eqn. (3.1), n =1000/(1-$10^{-4}10^{3}$) = 1000/0.9 =1111. Thus if the source strength is 1111 cps and dead time of the detector is 100 µs, then detector is dead for a period of 0.1 s in each second of its operation.
3. Calculate the maximum activity that could be counted using the detector given to you.

Experiment 3

STATISTICAL ASPECTS OF RADIOACTIVITY MEASUREMENTS

Radioactive disintegration is a random process. It is not possible to predict which atom will decay from an ensemble of atoms in a radioactive sample. Exact number of atoms that undergo decay in a given time duration cannot be predicted. Therefore, the results of a series of activity measurements of a sample containing a long lived radioisotope are expected to follow the Poisson or Gaussian distribution. For a Gaussian distribution the probability p_i of observing X_i events in a given time interval is given by

$$p_i = \frac{1}{\sqrt{2\pi\sigma^2}} \exp - \left[\frac{\left(X_i - \bar{X}\right)^2}{2\sigma^2} \right] \tag{3.8}$$

where $\bar{X}$ is the mean of the number of events and σ is the standard deviation. The standard deviation is the root mean square deviation of a large number of measurements. For a finite number of measurements (n), σ is given as:

$$\sigma^2 = \frac{\sum_{i=1}^{n} \left(X_i - \bar{X}\right)^2}{n-1} \tag{3.9}$$

The probability of a single observation being within $\pm\sigma$ of the mean is 68.3% and similarly 95.5% is the chance that a single observation has less than $\pm 2\sigma$ deviation from the mean. If a single measurement gives a value of X, there is a 68.3% chance that another measurement would lie between $X - \sigma$ and $X + \sigma$ and a 95.5% chance that a second measured value would lie between $X - 2\sigma$ and $X + 2\sigma$. The fact that radioactive decay obeys a random distribution law, it may be used to check the behaviour of the counting equipment on the assumption that any further deviation is due to erratic behaviour of the equipment.

Materials Required

GM counter set up, ^{137}Cs or any long lived source[1] and sample holder.

Procedure

1. Assemble the electronic units of GM counter set up and make connections as given in the instruction manual. Note down the settings.
2. Switch on the mains supply. After 10 minutes put on the 'EHT'. Set the EHT value to the operating voltage determined in Experiment 1. Allow to stabilize for half an hour.
3. Place the given source in the source holder and mount the source holder in a suitable groove of the sample stand.
4. Count for 1 minute or until about 10,000 counts are obtained and record the reading.
5. By fixing the counting time, take about 30 readings. Enter the data into the Table.

---

[1] The radioactive source has to be long lived such that it does not decay appreciably during the experiment.

Observation

Date	:				
GM Counter No.	:		GM tube No.	:	
Function	:	Time	Preset time	:	60 s
Paralysis time	:	250 µs	Operating voltage	:	V
Position	:	Groove No.	Sources	:	^{137}Cs

Table 3.3 - Counting Data

Sl. No.	Count rate (C_i)	Corrected count rate $X_i = (C_i - C_b)$	Deviation D $(X_i - \bar{X})$	$(X_i - \bar{X})^2$
1				
2				
3				
4				
5				
6				
7				
8				
9				
10				
11				
12				
13				
14				
15				
16				
17				
18				
19				
20				
21				
22				
23				
24				
25				
26				
27				
28				
29				
30				

Calculations

1. Calculate the average counts ($\overline{X}$) from the 30 readings.
2. Calculate the deviation from mean (X_i - $\overline{X}$) for each reading and enter in the Table.
3. Similarly calculate (X_i - $\overline{X}$)^2 for each reading and enter in the Table.
4. Calculate the standard deviation σ using the eqn. (3.9).
5. Calculate the mean deviation (d_m) using eqn. (3.10).

$$d_m = \frac{\sum\left|X_i - \overline{X}\right|}{n-1} \tag{3.10}$$

6. Calculate d_m/σ =
7. Calculate the number of readings in the range $\overline{X} \pm \sigma$ = (about 68.3% of total readings)
8. Find the number of readings in the range $\overline{X} \pm 2\sigma$ = (about 95.5% of total readings)
9. Obtain frequency p_i by grouping count rates. Plot p_i v/s X_i of each group. You will obtain a histogram which can be used to obtain a Gaussian plot. When the number of readings is very large, the plot will look like Fig 3.2.

Carry out the statistical tests as follows:

(i) Calculate the square root of the average values $(\overline{X})^{1/2}$ and verify whether this is nearly equal to the standard deviation (σ).

(ii) Calculate the ratio of mean deviation of the standard deviation i.e. d_m/σ. This should be nearly equal to 0.797.

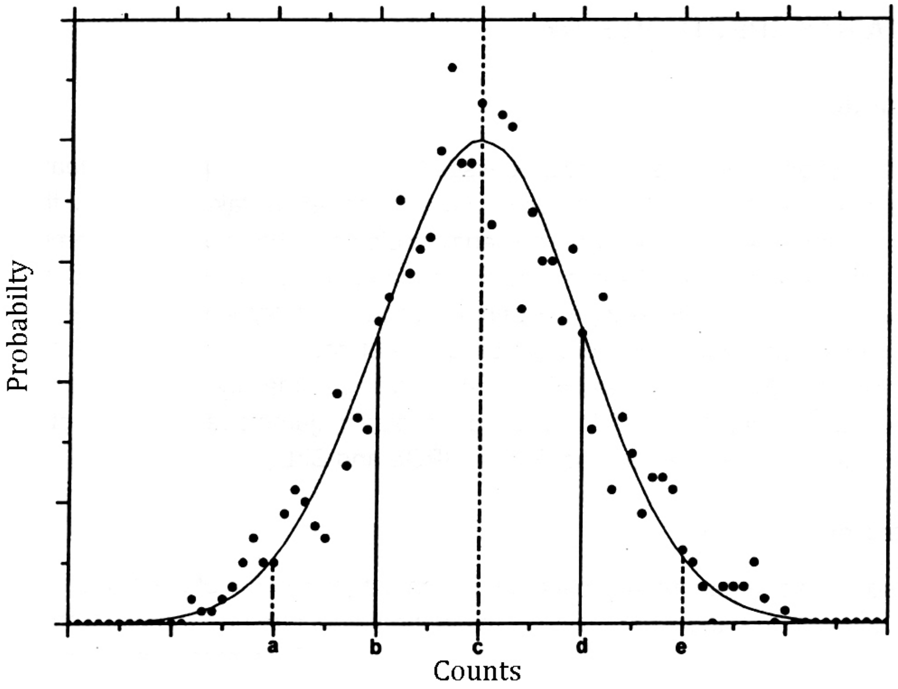


Fig 3.2 Gaussian distribution of counting data.
$a = \overline{X} - 2\sigma$, $b = \overline{X} - \sigma$, $c = \overline{X}$, $d = \overline{X} + \sigma$, $e = \overline{X} + 2\sigma$

2. GAMMA RAY SPECTROMETRY

2.1 Introduction

Nuclear reactions or nuclear decay processes (α or β emission) quite often leave a nucleus in an excited state. The de-excitation of such a nucleus generally takes place by the emission of gamma rays. Since the energy levels in a nucleus are discrete, the energy of the emitted photons is equal to the difference between the energies of two states of the nucleus involved in the transition, and is characteristic of the radioisotope. Gamma ray spectrometry is, therefore, a useful tool in the identification and assay of radionuclides. Two classes of detectors are used in gamma ray spectrometry: inorganic scintillators such as Nal(Tl) and semiconductor detectors such as HPGe. The oldest of these is the inorganic scintillator Nal(Tl). The high resolution gamma ray spectrometry is carried out using semiconductor detectors such as Ge(Li), HPGe and Si(Li).

Nal(Tl) Detector

The absorption of a gamma ray in a detector results in the migration of electrons from the valence band to the conduction band and leaving holes in the valence band. The electron and hole pairs move in the detector volume. Some of the holes combine with Tl atom to form Tl^+ which takes up an electron to form neutral Tl atom in an excited state, which in turn de-excites by emitting a photon in the visible region. Some of the photons reaching the photocathode of Photo Multiplier Tube (PMT) cause photoelectric effect and liberate electrons. These electrons are multiplied by a factor of about 10^6 by a series of dynodes inside the PMT. These electrons form the basic signal. At the anode, the charge collected across the RC coupling, produces a voltage pulse proportional in magnitude to the initial energy absorbed in the detector. On an average 100-300 eV of the absorbed energy is required for liberating an electron at photocathode surface and this results in the poor resolution of the detector (~7 to 10%).

A typical block diagram of a gamma ray spectrometric set up is shown in Fig.1.3. High voltage (EHT) in the range of 500 to 900 V is applied to PMT. One would expect a single peak in the spectrum corresponding to full energy if the gamma ray interacts in the detector by the photoelectric process only. A typical gamma ray spectrum of ^{137}Cs obtained using Nal(Tl) is shown in Fig. 3.3. A small peak seen at 75 keV is due to the X-rays of Pb which is used as a shielding material for the NaI(Tl) detector. When a gamma ray undergoes Compton scattering process, a fraction of the gamma ray energy is deposited in the detector and the rest of the energy is carried away by the scattered photon. Since the deposition of energy is incomplete and the extent of deposition depends on the scattering angle of the photon, a continuous response below the full energy peak is obtained.

When the gamma ray energy (E) is higher than 1.02 MeV ($2\ m_ec^2$), pair (electron + positron) production is an important mechanism for the energy loss of photons in matter. For momentum conservation electron and positron move in the opposite direction with equal kinetic energy corresponding to 0.5(E-1.02) MeV. After thermalisation, positron picks up an electron from the medium and gets annihilated producing two photons of 511 keV each. If both the annihilation photons are absorbed in the detector, then a photopeak corresponding to full energy of the gamma ray is observed. If one of the annihilation photons escapes from the detector, then a peak at E - 511 keV, called single escape peak is observed. If both the annihilation gamma rays escape the detector without interacting, then a photo peak corresponding to E - 2x511 keV, called double escape peak, is observed. If the pair production process takes place in materials outside the detector and one of the annihilation photons is absorbed by photoelectric process in the detector, then a peak at 511 keV is observed. It is called annihilation peak (c in Fig. 3.4). Probability of observing all these peaks increases with increasing energy of the gamma ray. A typical gamma ray spectrum of ^{60}Co obtained using NaI(Tl) is shown in Fig. 3.4.

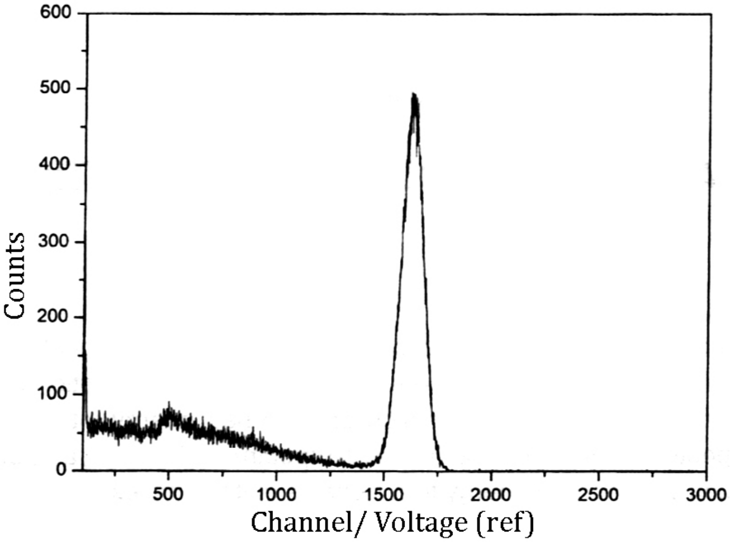


Fig 3.3 Gamma ray spectrum of ^{137}Cs obtained using NaI(Tl) detector

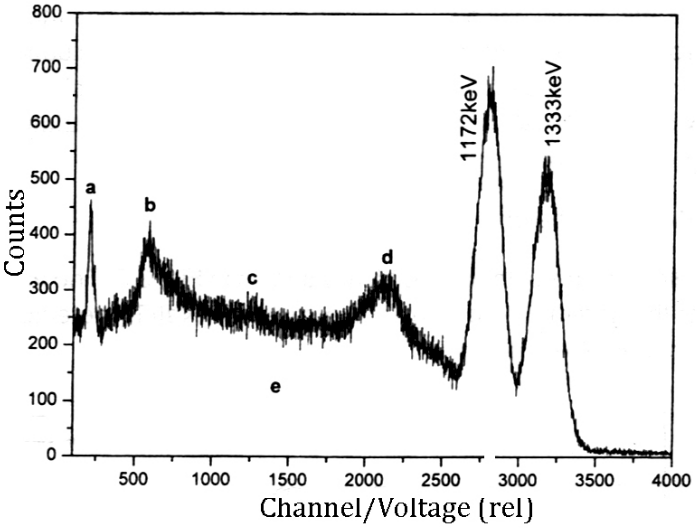


Fig 3.4 Gamma ray spectrum of ^{60}Co with NaI(Tl) detector, a=X-ray peak of lead, b= back scatter peak (255 keV), c = annihilation peak (511 keV), d = beginning of Compton edge and e = Compton continuum

Experiment 4

DETERMINATION OF RESOLUTION OF A NaI(Tl) DETECTOR

Discussion

Resolution of a detector is defined as its capability to resolve the signals due to two different gamma rays having energies which are close by. In the event of full energy absorption in the detector, and constancy of the factors involved in conversion of absorbed energy into electrical signal, it is expected that the spectrum observed should be a line spectrum. However, as discussed earlier, 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. In the case of a NaI(Tl) detector, it varies from 100 to 300 eV to produce a photoelectron at the photocathode of the PMT and in the case of a semiconductor detector it varies from 1.5 to 3.0 eV to produce an electron-hole pair. 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:

$$\% \text{ R} = 100 \text{ FWHM / Pulse height} \qquad (3.11)$$

The concept of resolution of a detector is graphically shown in Fig. 3.5. The values of FWHM are in the range of 7-10% of the gamma ray energy in the case of a NaI(Tl) detector and in the range of 0.1-0.2% in the case of a HPGe detector.

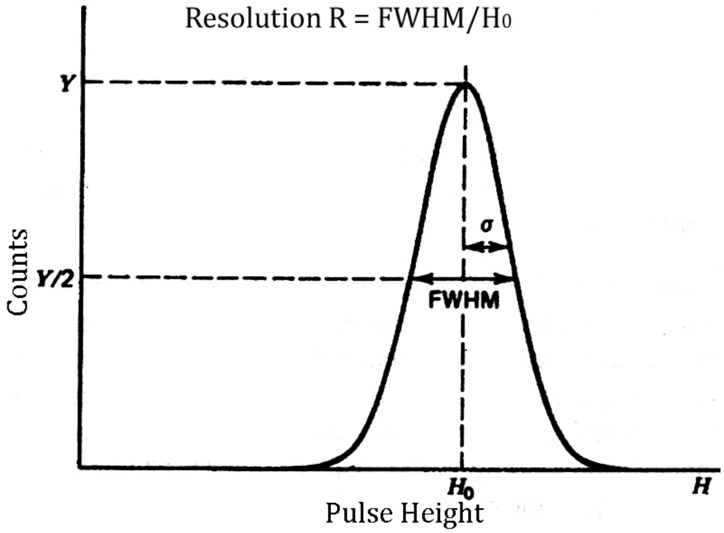


Fig 3.5 Definition of resolution of a gamma ray detector

In this experiment resolution of a Nal(TI) detector is determined. A photograph of Nal(TI) detector with a single channel analyser is shown in Fig. 3.6. The experiment is to be carried out using ^{137}Cs (662 keV) and ^{60}Co (1173 and 1332 keV) sources. Gamma spectra of ^{137}Cs and ^{60}Co are shown in Figs. 3.3 and Fig. 3.4 respectively.

Fig. 3.6 NaI(Tl) detector and single channel analyzer system.

The pulse height analysis is carried out using a single channel analyzer, wherein the pulses are sorted out in groups of energies, using a variable base line voltage (normally 0 to 10 V) and the window (normally set to 0.1 V).Thus at base line voltage V_B the pulses with heights between V_B and $V_B + 0.1$ V are allowed to reach the scalar. The count rate as a function of base line voltage is plotted to obtain the gamma ray spectrum of the source. The pulse height corresponding to the peak position (Vp) is proportional to the energy of the gamma ray and the full width at half maximum of the peak gives the resolution of the detector (Fig. 3.5). In this experiment V_p values corresponding to the three gamma ray energies are determined. As the pulse height is proportional to gamma ray energy, a plot of the peak voltage (Vp) against energy of the gamma ray will give the energy calibration, which is a straight line. The γ ray energy of unknown source can be determined using the calibration plot.

Materials Required

NaI(Tl) detector with accessories and single channel analyzer, radioactive sources: ^{137}Cs, ^{60}Co and unknown sources.

Procedure

1. Assemble the electronic units of NaI(Tl) detector and make connections as given in the instruction manual. Note down the settings.
2. Switch on the, mains supply. After 10 minutes put on the `EHT'. Raise the high voltage slowly until the rated value is reached. For 2" x 2" NaI(Tl) and the PMT, rated value of 'EHT'[2] is 700 V. Allow to stabilize for half an hour.
3. Adjust the base line voltage (V_B) to 0.2 volts. Fix the window width at 0.1 volt.
4. Place ^{137}Cs source in one of the grooves of the source stand in front of the detector and take the first reading.
5. Increase the base line voltage by 0.1 V and take the second reading. Repeat this by increasing V_B in steps of 0.1 V till the count rate first increases to a maximum and then falls to almost background level.
6. Plot the count rate as a function base line voltage + half of the window width. The peak Voltage (Vp) corresponds to the energy of 662 keV.
7. The FWHM of the peak corresponds to resolution in volts. Percentage resolution at 662 keV is obtained as FWHM/V_p x100.
8. Take out the source and place the empty source holder in the same grove of the stand, and repeat the steps 4 and 5 to obtain background counts (C_b). This will give the background spectrum in the chosen energy region.

---

[2] Ensure to apply EHT shown on the instrument

9. Repeat the procedure with ^{60}Co source and find out peak voltages corresponding to 1173 and 1332 keV gamma rays.
10. Obtain energy calibration plot taking energy on X-axis and peak voltage on Y-axis.
11. Repeat steps 4 to 7 with an unknown source after resetting V_B to 0.2 V.
12. Repeat step 8 to obtain the background spectrum corresponding to ^{60}Co γ peaks.
13. Find out the peak voltage for the unknown source and read out the energy from the calibration curve obtained in step 9.

Observations

Date

Detector	: NaI(Tl)	Detector No.	:
Amplifier Model	:	High voltage	: 700 V
Mode	: Differential[3]	Amplifier Gain	:
Base line	: As in Table 3.4	Window	: 0.1 V
Function	: Time & Operate	Sources	: ^{137}Cs, ^{60}Co & unknown
Position	: Groove No.	Preset Time	: 30 s

Table 3.4 - Counting data using ^{137}Cs source (γ energy : 662 keV)

Sl. No.	Base Line Voltage V_B	Counts/30 s	C_b 30 s	Sl. No.	Base Line Voltage V_B	Counts/30 s	C_b 30 s
1				13			
2				14			
3				15			
4				16			
5				17			
6				18			
7				19			
8				20			
9				21			
10				22			
11				23			
12				24			

---

[3] Counting Modes: When the full energy spectrum is obtained by scanning through a fixed window width (between two voltages) it is called differential counting. When the window is not used and the total count rate above a particular base line voltage is acquired, then it is called integral counting.

Table 3.5 - Counting data using ^{60}Co source (γ energies: 1172 & 1332 keV)

Sl. No.	Base Line Voltage V_B	Counts/30 s	C_b 30 s	Sl. No.	Base Line Voltage V_B	Counts/30 s	C_b 30 s
1				16			
2				17			
3				18			
4				19			
5				20			
6				21			
7				22			
8				23			
9				24			
10				25			
11				26			
12				27			
13				28			
14				29			
15				30			

Table 3.6 - Counting data using unknown source

Sl. No.	Base Line Voltage VB	Counts/30 s	Cb 30 s	Sl. No.	Base Line Voltage VB	Counts/30 s	Cb 30 s
1				13			
2				14			
3				15			
4				16			
5				17			
6				18			
7				19			
8				20			
9				21			
10				22			
11				23			
12				24			

Calculations

1. Plot the graph between the count rate (Y-axis) and base line voltage (X-axis) for ^{137}Cs and ^{60}Co sources. Your plots will look similar to Figs. 3.4 and 3.5, respectively.
2. Find the resolution of the detector for 662 keV of ^{137}Cs and for 1172 and 1332 keV of ^{60}Co. % resolution = FWHM/V_p x 100.
3. Obtain the energy calibration curve by plotting energy on X-axis and peak voltage on Y-axis.
4. Convert the value of FWHM obtained in volts into energy (keV) using the calibration curve. Calculate $\Delta E/\Delta V$ in keV/volts.
5. Comment on the ability of the detector to resolve these two ^{60}Co peaks.
6. Plot the graph between the count rate and base line voltage for the unknown source as in step 1.
7. Identify the peak position(s) and note the base line voltage(s) corresponding to the peak position(s).
8. Identify the energy(ies) of the unknown gamma ray(s) from the calibration curve.

Experiment 5

DETERMINING THE ACTIVITY OF A GAMMA SOURCE

Discussion

The absolute detection efficiency, ε, of a detector is the ratio of counts under the photopeak corresponding to full energy of a particular gamma ray to the total number of gamma rays of that energy that are emitted by the source. ε is a product of the intrinsic efficiency of the detector and geometrical efficiency. Therefore, for a given gamma ray energy, in a fixed source to detector geometry, the efficiency is a constant. Abundance of a gamma ray (a), detection efficiency (ε), and the counts (C) observed under photopeak corresponding to energy E are related as:

$$C = A\, \varepsilon\, a \tag{3.12}$$

where A is the disintegration rate of the radioisotope. It should be noted that units of C are counts per second (cps) if A is expressed in disintegrations per second (dps).

For measuring the strength of an unknown source of a radioisotope, a similar source having a known strength is prepared and counted in a system along with the unknown source. The ratio of the activities of two sources of the same radioisotope, therefore, is equal to the ratio of the areas under their respective photopeaks. The activity A_x of an unknown source is thus given as:

$$A_x = (C_x / C_s)\, A_s \tag{3.13}$$

where C_x and C_S are the photopeak areas (in cps) corresponding to the gamma rays of the unknown source and standard source respectively, and A_s is the absolute activity of the standard source. In this experiment, the activity of a sample of unknown strength of ^{137}Cs is determined by using the activity of a standard and the ratio of the peak areas of unknown and standard sources of ^{137}Cs.

Materials Required

Nal(TI) detector set up with accessories and SCA, sample stand and ^{137}Cs source and sources of known and unknown strengths.

Procedure

1. Assemble the electronic units of NaI(Tl) counting setup and make connections as given in the instruction manual. Note down the settings.
2. Put on the mains supply. After 10 minutes put on the 'EHT'. Raise the high voltage slowly until the rated values is reached. Allow to stabilize for half an hour.
3. Calibrate the set up using ^{137}Cs and ^{60}Co as described in the Experiment 4. From the plot of ^{137}Cs spectrum, note down the base line voltage at the start of the peak (V_1) and end of the peak (V_2). Keep the window width equal to V_2-V_1 and base line at V_1.
4. Mount the ^{137}Cs standard source in one of the shelves of the source holder. Count this source for sufficiently long time. The counts observed is the integrated peak area starting at V_1 and ending at V_2. Calculate cps (C_S).
5. Clear the previous data and place the ^{137}Cs source of unknown activity in place of the standard. Count this source also for a sufficiently long time. Calculate the cps under the photo peak (C_S).
6. Remove sources to a distance, clear data and obtain background count for 1000 seconds under the same settings. Calculate the background cps (C_b).

Observations

Date

Detector	:	NaI(Tl)	Detector No.	:	
Amplifier Model	:		High voltage	:	700 V
Mode	:	Differential	Amplifier Gain	:	3.0 V
Base line	:	V	Window[4](V_2-V_1)	:	V
Function	:	Time & Operate	Sources	:	^{137}Cs
Position	:	Groove No.	Preset Time	:	s
Background[5] Counts/1000s	:		Background Counts/ 60s C_b	:	

Table 3.7 - Measured counting data

Sl. No.	Sample	Integral peak area cps	Average cps	Background Corrected cps
1	Standard			
2	Standard			
3	Unknown			
4	Unknown			

Calculations

1. Calculate A_x the activity of the unknown using eqn. (3.13).
2. You are given two ^{131}I sources (E= 364 keV) of a known and an unknown activity. Use the above procedure to determine ^{131}I activity in the unknown sample.

---

[4] By setting the base line at the beginning of the peak V_1, and window at V_2-V_1 all counts under the 662 peaks get recorded and, therefore, these counts are a measure of the peak area.

[5] Background counts obtained here are integral counts in the chosen window. Correct practice is to obtain a pulse height spectrum, mark the counting minima on both the sides of peak centroid and subtract trapezoidal area from the integral peak area.

Experiment 6

DETERMINATION OF ABSOLUTE ACTIVITY BY HIGH RESOLUTION GAMMA RAY SPECTROMETRY WITH HIGH PURITY GERMANIUM (HPGe) DETECTOR

Discussion

The semiconductor detectors such as Ge(Li) and high purity germanium (HPGe) are used for high resolution gamma ray spectrometric measurements. Germanium detectors have resolution of the order of 1.0 to 2.5 keV in the energy range of 100 to 2500 keV. Due to their excellent resolution, HPGe detectors are widely used in measuring the complex gamma ray energy spectra of radioactive samples containing many radioisotopes from these spectra activities due to individual constituents can be obtained. A multichannel analyser (MCA) differentiates the incoming signal into peaks having different energies. To arrive at absolute activities from measured count rates, the efficiency of the detection system at different peaks (energies) has to be determined. The efficiency of detection decreases as a function of energy, similar to the cross section for photoelectric absorption as shown in Fig. 3.7. A photograph of the detector and MCA is shown in Fig. 3.8. Photopeak count rate (C) under a gamma ray of particular energy E, is directly proportional to the activity of the source and is given by eqn. (3.12).

$$C = A\,a\,\varepsilon \qquad (3.12)$$

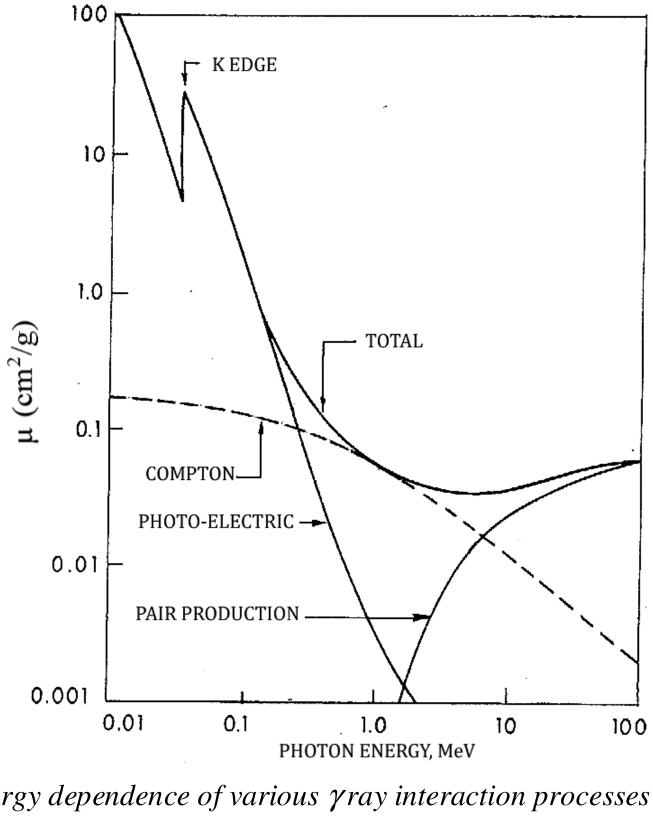


Fig 3.7 Energy dependence of various γray interaction processes in materials.

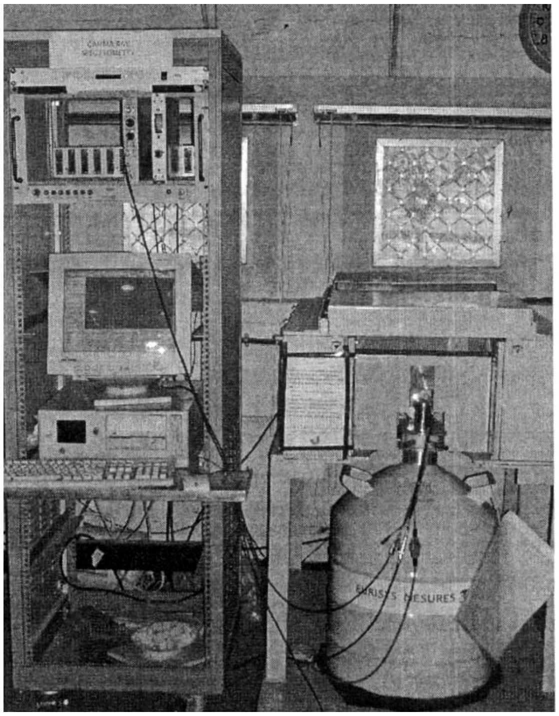


Fig 3.8 Gamma ray spectrometer consisting of HPGe detector, accessories and a PC based MCA

Material Required

High Purity Germanium (HPGe) detector coupled to a Multi Channel Analyzer (MCA), radioactive sources: ^{241}Am, ^{152}Eu, ^{137}Cs, ^{60}Co, and unknown sources.

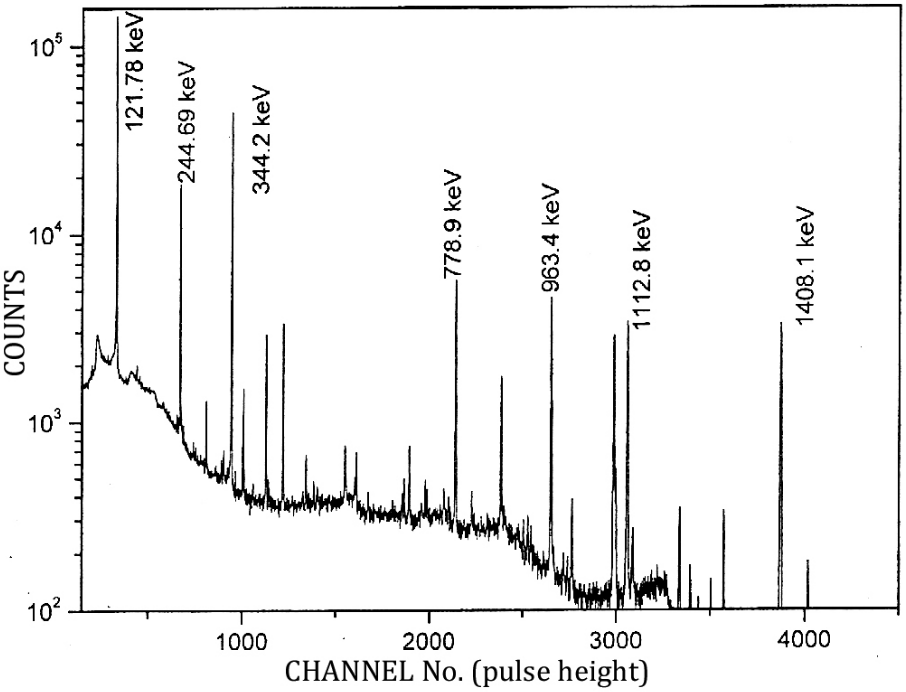


Fig 3.9 Gamma Ray Spectrum of ^{152}Eu acquired on an MCA using HPGe Detector

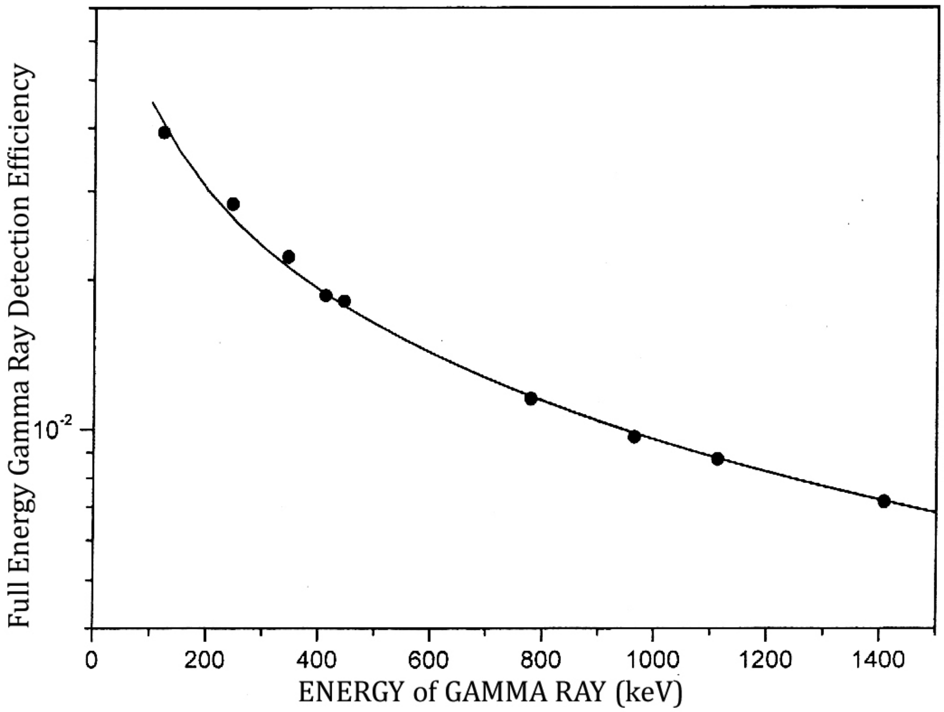


Fig 3.10 Detection Efficiency of HPGe Detector as a Function of Gamma Ray Energy

Procedure

1. Fill the dewar of the HPGe detector with liquid nitrogen. The detector should be cooled for 6 hours or more before high voltage is applied.
2. Assemble the electronic units, HPGe detector and MCA. Make sure that the connections are made as given in the instruction manual. Note down the settings.
3. Switch on the mains supply. After a few minutes, slowly apply high voltage in steps of 100 volts until rated voltage is reached. Allow to stabilize for an hour.
4. Set the appropriate value for lower level discriminator (LLD) (around 20 mV).
5. Mount ^{241}Am source in a shelf of the sample holder. Acquire spectrum for 1000 s. Use the same shelf for counting all other sources.
6. From the spectrum, obtain the channel numbers corresponding to each photo peak maximum.
7. Repeat this with other sources and tabulate the results.
8. Place the standard source of ^{152}Eu in a shelf of sample holder of HPGe detector. Acquire the γ-spectrum for a period of 1000 s and record the spectrum. The γ-ray spectrum is expected to look like Fig. 3.9.
9. Remove the source and acquire background spectrum for 2000 s.

Observations

Date

Detector	: HPGe	Detector No.	:
Amplifier Model	:	High Voltage	: V
LLD	: Mv	MCA Mode	: Pulse Height Analyzer
Amp. Coarse Gain	:	Amp. Fine Gain	:
Time Constant	:	Calibration Standard	: ^{152}Eu
Shelf Number	:	Sources	: ^{241}Am, ^{137}Cs, ^{60}Co

Table 3.8 - Peak positions for energy calibration

Sl. No.	γ ray Source	Peak Energy E (keV)	Centroid Channel No.
1	^{241}Am	60	
2	^{137}Cs	662	
3	^{60}Co	1172	
		1332	

Calculations

A. Identification of gamma ray energy of unknown source

1. Obtain a calibration graph by plotting channel number on X-axis and the corresponding energy on Y-axis. It should be a straight line of the type E = mX + c, where X is the channel number corresponding to energy (E) and, m and c are slope and intercept respectively. Calculate the calibration constants m and c: m = ……………..keV/channel and c = ……………..keV
2. Determine the peak positions from the spectrum obtained using unknown source.
3. From the calibration plot, note the energy corresponding to the channel for each peak of the unknown source. It may be noted that most of the MCA systems have built in programmes for energy calibration.

Source strength of ^{152}Eu (A) : dps on :

Counting time : s

Table 3.9 - Peak areas for different gamma ray peaks of ^{152}Eu

Energy keV	γ abundance (a)	Channel No.	FWHM keV	Peak area counts	Efficiency
121.78	0.282				
244.69	0.074				
344.20	0.264				
778.90	0.130				
963.40	0.145				
1112.80	0.135				
1408.10	0.207				

B. Determination of detection efficiency of HPGe detector as a function of γ-ray energy.

1. Calculate peak areas corresponding to different gamma rays of ^{152}Eu. Record the channel number, area and FWHM for each peak in Table 3.9.
2. Calculate efficiency using the relation between dps and cps: $\varepsilon = C/A\ a$ (Note: Calculate the dps as on the date of experiment using decay correction).
3. Plot efficiency ε as a function of energy E in keV on a semilog graph paper taking ε on Y-axis and E on X-axis. Your plot will look like Fig. 3.10.
4. Calculate efficiency values at 411.1 and 867.3 keV of ^{152}Eu from the calibration curve.

5. Calculate the gamma ray abundances for the peaks at 411.1 keV and 867.3 keV of ^{152}Eu. Your values should be 0.022 and 0.042 respectively.
6. Find full width at half the maxima (FWHM) for at least four peaks and compare the values with those obtained using NaI(TI) detector set-up.
7. Fit the efficiency (ε) and energy (E) using eqn. 3.14. Compute the efficiency values corresponding to 411.1 and 867.3 keV. Compare these values with those obtained in step 4.

$$\ln \varepsilon = c_1 + c_2 \ln(E) + c_3 (\ln(E))^2 \tag{3.14}$$

where c_1, c_2, c_3 are constants.

Experiment 7

THE ABSORPTION COEFFICIENT AS A FUNCTION OF GAMMA RAY ENERGY

Discussion

Absorption of gamma rays in matter leads to a decrease in the intensity of transmitted gamma rays. When a foil of thickness x is transposed between a source of strength I_0 and the detector (Fig. 3.11), the transmitted intensity I is related by Lambert's Law:

$$I = I_0 \, e^{-\mu x} \qquad (3.15)$$

where μ is the linear absorption coefficient in cm^{-1} and x is the thickness of the absorber in cm. The absorption of γ-rays depends on the atomic number of the absorber and the gamma ray energy. A typical plot of I vs x is also given in Fig. 3.11.

The units of μ and x should have inverse relation so that the product is a dimensionless quantity. Very often mass absorption coefficient is used in practice. The mass absorption coefficient (μ_m) is obtained by dividing μ with density (ρ) of the absorber. The thickness 'x' is multiplied with ρ to obtain x_m such that the product $\rho_m.x_m$ remains a dimension less quantity. Thus the unit of mass attenuation coefficient μ_m is cm^2/g and x_m is g/cm^2. Mass absorption coefficients for ^{60}Co, ^{54}Mn, ^{137}Cs and ^{57}Co in lead absorbers are given in Table 3.10

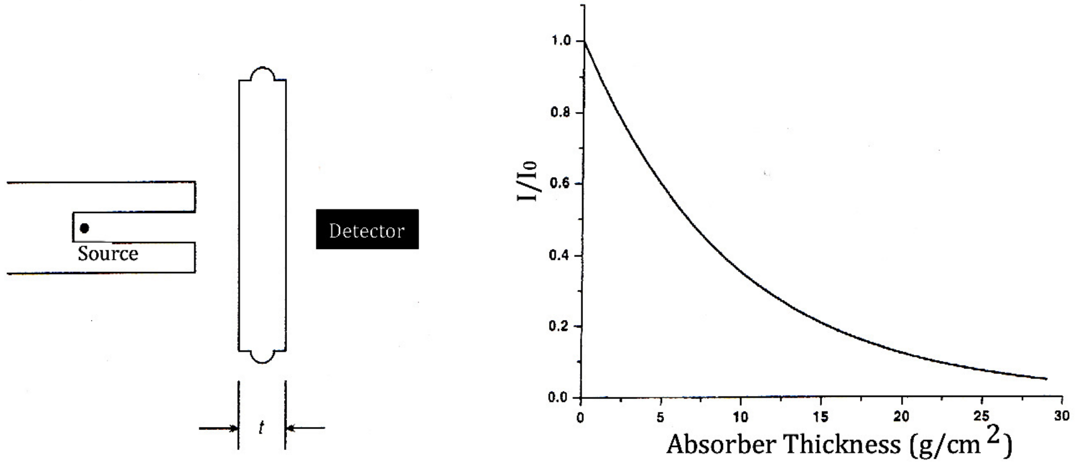


Fig 3.11 Schematic of γ ray absorption experiment and intensity variation as a function of absorber thickness

Table 3.10 - Mass absorption coefficients for ^{60}Co, ^{54}Mn, ^{137}Cs and ^{57}Co γ rays in lead.

Isotope	Energy (MeV)	μ_m (lead) cm^2/g
^{60}Co	1.17, 1.33 (average 1.25)	0.063
^{54}Mn	0.835	0.083
^{137}Cs	0.662	0.105
^{57}Co	0.122	3.100

In this experiment, mass attenuation coefficient for 0.662 MeV and 1.25 MeV gamma rays would be determined using Pb and Al absorbers.

Materials Required

^{137}Cs and ^{60}Co sources, NaI(Tl) counting setup with accessories and absorber sets of Pb and Al.

Procedure

1. Make all connections of NaI(Tl)) detector as given in the instruction manual. Put on mains and after 10 minutes apply high voltage slowly until rated value is reached. Allow it to stabilize for 30 minutes.
2. Obtain γ spectrum of ^{137}Cs by using NaI(Tl) with a single channel analyzer (as in Experiment 4).
3. Use this spectrum to decide the base line voltage and window width for measuring the peak area for 662 keV photons (as described in Experiment 5).
4. Place ^{137}Cs source and the absorber holder without absorber on a shelf of the sample holder. Count for 100 seconds and note the reading.
5. Place the first lead absorber (0.9 g/cm^2) on the absorber holder without disturbing the source. Count for 100 seconds and note the reading.
6. Repeat step 5 for the rest of the lead absorbers and combinations of same to give a total thickness of at least 13 g/cm^2. This will give a total of about 8 data points.
7. Repeat steps 5 and 6 for Al absorber sets.
8. Acquire background counts for 300 s.
9. Repeat steps 2 to 3 to obtain the γ-ray spectrum of ^{60}Co.
10. Repeat steps 4 & 5 to obtain data for mass attenuation coefficient using Al absorbers. Enter the data in Table 3.13
11. Repeat steps 4 & 5 to obtain data for mass attenuation coefficient using Pb absorbers. Enter the data in Table 3.14.

Observations

Date

Detector	:	NaI(Tl)	Detector No.	:	
Amplifier Model	:		High voltage	:	700 V
Mode	:	Differential	Amplifier Gain	:	3.0
Base line	:	V_1 Volts corresponding to each peak	Window (^{137}Cs)	:	V_2-$V_1$for each peak
			Window (^{60}Co)	:	-do-
Function	:	Time & Operate	Sources	:	^{137}Cs and ^{60}Co
Position	:	Lowest groove	Preset Time	:	s
Background Counts/300s (^{137}Cs)	:		Background count/100 s (C_b)	:	
Background Counts/300s (^{60}Co)	:		Background count/100 s (C_b)	:	

Table 3.11 - Data for attenuation of γ-radiation from ^{137}Cs in lead (Density :11.34 g cm^{-3})

Sl. No.	Pb absorber g cm^{-2} (d)	Observed Counts /100 s (C_0)	Corrected Counts (C_c = (C_0- C_b)
1			
2			
3			
4			
5			
6			
7			
8			
9			
10			
11			
12			
13			
14			

Table 3.12 - Data for attenuation of γ-radiation from ^{60}Co in lead (Density :11.34 g cm^{-3})

Sl. No.	Pb absorber g cm^{-2} (d)	Observed Counts /100 s (C_0)	Corrected Counts (C_c = (C_0- C_b)
1			
2			
3			
4			
5			
6			
7			
8			
9			
10			
11			
12			
13			
14			

Table 3.13 - Data for attenuation of γ-radiation from ^{137}Cs in aluminium (Density: 2.7 g cm^{-3})

Sl. No.	Pb absorber g cm^{-2} (d)	Observed Counts /100 s (C_0)	Corrected Counts ($C_c = (C_0 - C_b)$
1			
2			
3			
4			
5			
6			
7			
8			
9			
10			
11			
12			
13			
14			

Table 3.14 - Data for attenuation of γ-radiation from ^{60}Co in aluminium (Density: 2.7 g cm^{-3})

Sl. No.	Pb absorber g cm^{-2} (d)	Observed Counts /100 s (C_0)	Corrected Counts ($C_c = (C_0 - C_b)$
1			
2			
3			
4			
5			
6			
7			
8			
9			
10			
11			
12			
13			
14			

Calculations

1. Make a plot on a linear graph paper taking the peak area (corrected count) on Y-axis and absorber thickness on X-axis.
2. From the above plot, determine the half value layer (HVL) for lead absorber. The HVL is defined as the thickness of material that will decrease the gamma ray intensity to one-half of its original value.
3. From eqn. (3.15), $I/I_0 = 0.5$ for the (HVL). Therefore, μ_m can be determined from the following equation
   $\mu_m = 0.693/\text{HVL cm}^2/\text{g}$ (3.16)
   The value of μ_m for lead for 662 keV of ^{137}Cs is 0.105 cm^2/g. The measured value should be within 10% of this value.
4. Repeat the calculations for ^{60}Co source.
5. Estimate the mass attenuation coefficient values for lead at 662 keV and at 1250 keV (mean of 1173 + 1332).
6. Carry out similar calculations for aluminium absorbers.

Comment

This experiment can be used gainfully to determine the thickness of thin foils of any material. A calibration plot has to be obtained between intensity (I) and thickness of the foil of the same material. By measuring the intensity after passing through the unknown foil and using the calibration plot, thickness of the unknown foil is determined.

Experiment 8

BETA BACKSCATTERING AS A FUNCTION OF ATOMIC NUMBER

Discussion

Beta particles undergo multiple scattering while passing through a medium. Their path, inside the medium, is torturous as shown in Fig. 3.12. The extent of scattering depends upon density (ρ) and atomic number of the medium, and the β energy. In this process some of the β-particles get scattered back in the direction of the source. Measurement of this backscattered radiation is widely used for measuring thickness of foils/coatings etc. For a β source, deposited on a plate, the count rate observed in a counter could be more than 50% of the source strength expected from the 2-π geometry. In fact, increasing the thickness of the backing enhances the count rate due to enhanced backscattering.

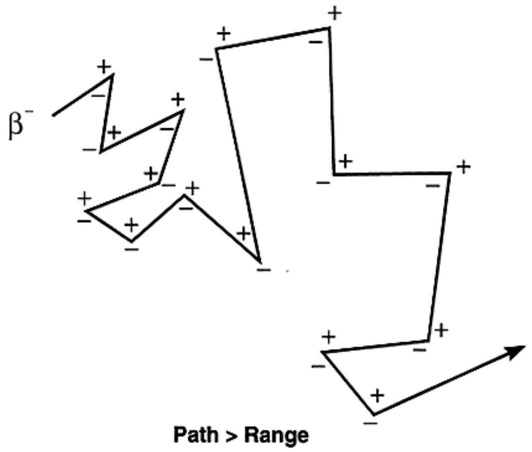


Fig. 3.12 Path of beta particles from a source in a medium.

The measured count rate (C), is related to the disintegration rate of the radioisotope (A), as follows:

$$C = A\, a_s\, a_B\, \Omega\, a_W\, \varepsilon_\beta \qquad (3.17)$$

where a_s is the self absorption fraction accounting for absorption of β radiations in the source, a_B is the back scattering fraction accounting for the β radiations that are scattered by the source backing in the direction of the detector, Ω is the solid angle subtended by the source at the window of the counter, a_W is the fraction of the absorption of radiation in the window and ε_β is the intrinsic efficiency of the detector used. All these factors depend on the materials used for backing and window, as well as the energy of the β particle. This experiment is designed to measure backscattering fraction and its effect on the observed count rate.

Materials Required

GM counter set up, sample holder, radioactive source ^{32}P and metal sheets.

Procedure

1. Assemble the electronic units of GM counter set up and make connections as given in the instruction manual. Note down the settings.

2. Switch on the mains supply. After a few minutes put on the 'EHT'. Allow to stabilize for half an hour.
3. Place the beta source (on a thin Perspex plate) in a shelf of the sample holder of the GM counter. Perspex being a low density and low Z material has a very small backscatter fraction. Count the activity for 60 seconds.
4. Place an aluminium backscattering sheet in contact with the source at the back of Perspex plate. Count for 60 seconds and record the reading.
5. Repeat this using sheets of identical thickness of the other materials.

Observations

Date

GM Counter No.	:		GM tube No.	:	
Function	:	Time	Preset time	:	60 s
Paralysis time	:	250 µs	Operating voltage	:	V (from Experiment 1)
Position	:	Groove No.	Source	:	^{32}P
Background counts/1800s	:		Background count/60 s (C_b)	:	

Table 3.15 - Data for beta backscattering analysis

Sl. No.	Material	Atomic Number	Net counts, cpm	Backscattering Factor
1	Perspex			
2	Aluminium			
3	Iron			
4	Copper			
5	Cadmium			
6	Molybdenum			
7	Tungsten			
8	Lead			

The Backscattering Factor (BF) is defined as the ratio of counts/minute with a scattering medium, divided by the counts/minute without a scattering medium. For Perspex we can presume BF to be unity and calculate BF for other materials based on the counts observed with Perspex backing. Calculate this value for each measurement and fill in the table. It would be noticed that BF will increase with increasing value of ρ and Z. Calculate the percent backscattering from the data and plot this value (Y-axis) as a function of the atomic number (X-axis) of the backscattering material.

Experiment 9

BETA ENERGY DETERMINATION BY FEATHER'S ANALYSIS

Discussion

Spectroscopic methods. are used to determine the alpha, beta and gamma energies. However, more conveniently, radiation energy is often obtained by determining the range of radiations in a medium like aluminium. A GM counter can be used to determine the range of beta particles. The absorption curve is obtained by plotting the count rate on Y-axis and the thickness (mg cm^{-2}) of absorbers on X- axis of a semi-log paper. In beta decay, the energy is shared between the β-particle and the neutrino and accordingly β-particle energy spectrum is continuous between 0 and the maximum energy, β_{max} (Fig. 3.13). Experimentally it is observed that absorption of β- particles, of a given β_{max} nearly follows the exponential law. Some radioisotopes have a more complex decay mode and emit more than one group of β-particles having different β_{max}. In such cases the shape of the absorption curve is altered. A tailing at the end of the range is obtained if the radioisotope also emits gamma rays. In such cases extrapolated range has to be used to obtain the corresponding beta energy. Ranges obtained by extrapolation are called 'visual ranges' and are often associated with large errors. By using the Feather's method, a better estimate of range/energy is obtained.

Feather's analysis is a comparison method. In this method, absorption curve of a beta emitter of unknown energy is compared with that of a standard beta emitter. Additionally, and most importantly, the tail part of the absorption curve is not used in the analysis.

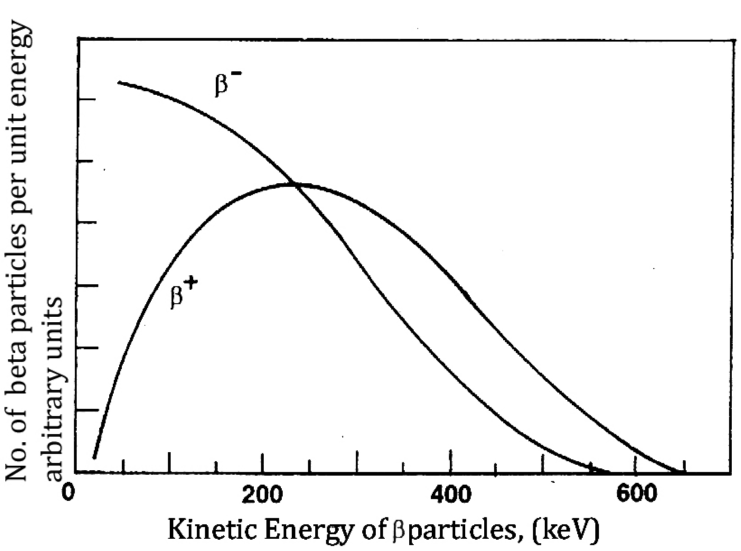


Fig 3.13 Beta energy spectrum of ^{64}Cu

The absorption curve of a pure beta emitter like ^{32}P (range in aluminium = 785 mg cm^{-2}) is measured and divided into 10 equal parts to obtain activity corresponding to fractional ranges of 0.1, 0.2 ... 1.0. These activities are marked on Y-axis and transferred onto a strip of linear paper. This marked paper is known as Feather analyzer. By super imposing the Feather analyzer on the absorption curve measured using the unknown source like ^{35}S or ^{210}Bi, under conditions identical to those of the standard, visual ranges corresponding to fractional ranges of 0.1,0.2 ... 0.7 are obtained. From these, apparent ranges are calculated by dividing the fractional ranges by the fraction. When apparent ranges are plotted (Y-axis) against the fractional ranges (X-axis), a smoothly varying curve is obtained as in Fig.3.14. The range of unknown β energy is obtained by extrapolating this curve to 1.0. From the range energy relations, energy of the unknownβ-emitter (β_{max}) is calculated.

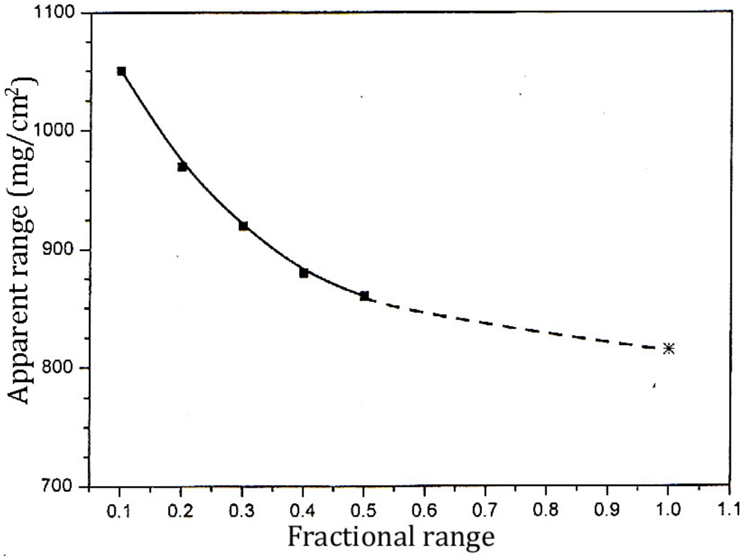


Fig 3.14 A plot of apparent range v/s fractional range for determination of β_{max}.

Materials Required

GM counter set up with accessories, sample stand with shelves, radioactive sources ^{32}P, ^{35}S and ^{210}Bi and aluminium absorbers set.

Procedure

1. Assemble the electronic units of GM counter set up and make connections as given in the instruction manual. Note down the settings.
2. Switch on the mains supply. After 10 minutes put on the `EHT'. Allow to stabilize for half an hour.
3. Count the background for 30 min.
4. Place the ^{32}P source facing the window in the shelf marked for source. Count for an hour or a suitable period so as to get about 10,000 counts. Note the time and counts. Calculate the count rate.
5. Place an aluminium absorber before the source and count as in step 4.
6. Repeat the counting by placing absorbers of different thickness as in step 4.
7. Tabulate all the data.
8. Replace ^{32}P source with unknown source whose β energy has to be determined.
9. Repeat steps 4 to 7 with unknown source.

Observations

Date					
GM Counter No.	:		GM tube No.	:	
Function	:	Counts	Preset time	:	10,000 (to obtain good statistics)
Paralysis time	:	250 µs	Operating voltage	:	V (from Experiment 1)
Position	:	Groove No.	Source	:	^{32}P and unknown source
Background counts/1800s	:		Background count/60 s (C_b)	:	

Table 3.16 - Counting data using ^{32}P source

Sl. No.	Absorber mg cm^{-2}	Counting Time s	Counts C	Count Rate cps	Corrected count rate (cps-C_b)
1					
2					
3					
4					
5					
6					
7					
8					
9					
10					

Table 3.17 - Counting data for unknown source

Sl. No.	Absorber mg cm^{-2}	Counting Time s	Counts C	Count Rate cps	Corrected count rate (cps-C_b)
1					
2					
3					
4					
5					
6					
7					
8					
9					
10					

Calculations

1. Plot the absorption curve for ^{32}P on a semi log paper taking the count rate (C) on Y-axis and thickness of the absorber on X- axis.
2. Divide the range into the fractions corresponding to 0.1, 0.2, 0.3 ...1.0. Mark the corresponding count rate on Y-axis and transpose the values onto a linear strip, known as Feather analyzer.
3. Plot the absorption curve for unknown as in step 1.
4. Keep the Feather analyzer along the Y-axis and mark the positions corresponding to fractional values of 0.1, 0.2, 0.3...

5. Note the corresponding values of thickness of the absorber (R_i) from X-axis of unknown absorption curve.
6. Divide R_i by corresponding fraction to obtain apparent ranges and note them.
7. Plot apparent range curve on a linear graph by taking apparent range on Y-axis and fractional range on X-axis. Your plot should be similar to Fig .3.14. Extrapolate X value to 1.0 and take the corresponding range value. This is the range of R particles of unknown energy.
8. Convert this range into energy (β_{max}) using range energy relation.

3. SOLID STATE NUCLEAR TRACK DETECTOR (SSNTD) TECHNIQUE

Experiment 10

ESTIMATION OF ALPHA ACTIVITY USING SSNTD

Discussion

Solid State Nuclear Track Detectors (SSNTD) are very useful to measure activity of the radioisotopes that undergo α-decay or spontaneous fission. Charged particles like α-particles while passing through films of insulators like CR-39 damage the material at the site they interact. By careful etching with alkali or acid, the damages appear as pits which are called nuclear tracks. The number of tracks are a direct measure of the activity of the radioisotopes that are emitting α-particles. Alpha activity in matrices like environmental samples and mineral samples could be measured with high reliability. Thin films of mica, lexan and CR 39 are some of the insulators that are used as solid state nuclear track detectors. Alpha activity originating from solids and liquids can be measured using SSNTD. Track density T_d, (number of tracks/cm^2), is directly related to the alpha activity in Bq/mL (D_s) by equ. 4.18

$$T_d = K_{wet} D_S t \qquad (4.18)$$

where K_{Wet} is the track registration efficiency in liquids and t is the exposure time. For CR-39, K_{wet} is about 5×10^{-4} cm. Determination of alpha activity in liquid samples is described below.

Materials Required

Uranium nitrate solution (~100 mg/mL), graduated pipette, CR-39 film, 100 mL beakers, constant temperature bath, burner, 6M NaOH solution, thermometer, counter and 500X optical microscope.

Procedure

1. Prepare a stock solution of 6N NaOH.
2. Transfer 250 mL of NaOH solution to heating bath and heat it to a steady temperature of 70°C
3. Keep a CR-39 track detector in contact with the 10 mL of uranium nitrate solution for 1-2 h.
4. Etch the exposed CR-39 film for 3-4 h in alkali bath prepared in step 2, wash it for 10 ruin under running water and wipe it dry.
5. Observe the film under the microscope at a fixed magnification between 400 to 500.
6. Count the tracks in the same view. Repeat observations by taking 5-10 different views (the reliability of the results is improved by using larger number of scans).
7. Calculate the area of view using a stage micrometer.

Observations

Date

Optical microscope used : Magnification :

Area of the view (field) :

Table 3.18 - Observed track data

Sl. No.	No. of tracks
1	
2	
3	
4	
5	
6	
7	

Calculations

1. Take the average track density (Number of tracks per cm^2)
2. Calculate the total alpha activity using the eqn. 4.18.
3. Compare this value with that obtained by radiometric assay of uranium nitrate solution.
4. Repeat this experiment with an environmental sample

Comments

1. What will happen if there are more than one alpha decaying isotope in the sample?

Experiment 11

DETERMINATION OF ALPHA TO FISSION BRANCHING RATIO IN THE DECAY OF ^{252}Cf

Discussion

Californium-252 decays by alpha emission as well as by spontaneous fission, the respective partial half lives being 2.64 y and 85y. The alpha to fission branching ratio can be measured using an SSNTD since the LET of fission fragments and alphas are distinctly different. The size of the nuclear tracks produced by fission fragments are substantially larger compared to the tracks due to alphas. The objective of the present experiment is to determine the total alphas emitted by ^{252}Cf source and also to determine the ratio of alphas to fission fragments to arrive at the branching ratio.

Materials Required

An electrodeposited source of ^{252}Cf on 25 mm dia. SS planchette (source activity approximately 5000 dpm (alpha) and source diameter ~10 mm), an etching bath, NaOH pellets, stirrer, pieces of plastic film CR-39 as track detectors, thermometer and an optical microscope.

Procedure

1. Prepare a 6N NaOH solution.
2. Transfer 250 mL of 6N NaOH to the heating bath and heat it to a steady temperature of 70°C.
3. Keep a CR-39 track detector in contact with the ^{252}Cf source for 20-30 min.
4. Etch the exposed CR-39 film for 3-4 h in the alkali bath (prepared in step 2), wash it for 10 min under running water and wipe it dry.
5. Observe the film under the microscope at a fixed magnification between 400 to 500. The large sized tracks are due to fission fragments and smaller ones are due to alpha particles.
6. Count each type of tracks in the same view. Repeat observations by taking 5-10 different views (the reliability of the results is improved by using larger number of scans).
7. Calculate the area of view using a stage micrometer.

Observations

Date:

Optical microscope used : Magnification:

Area of the view (field) :

Table 3.18 - Observed track data

Sl. No.	No. of large Tracks	No. of small Tracks
1		
2		
3		
4		
5		
6		
7		

Calculations

1. Calculate the alpha and fission fragment track density. (per cm^2) from the above data.
2. Calculate the ratio of the number of alpha tracks to the number of fission tracks.
3. Express the ^{252}Cf source strength in dps per cm^2 based on the following:
   (a) condition of exposure: 2π geometry
   (b) alpha mode of decay = 96.9%, Spontaneous fission mode = 3.1%, Number of fission fragments/fission =. 2.

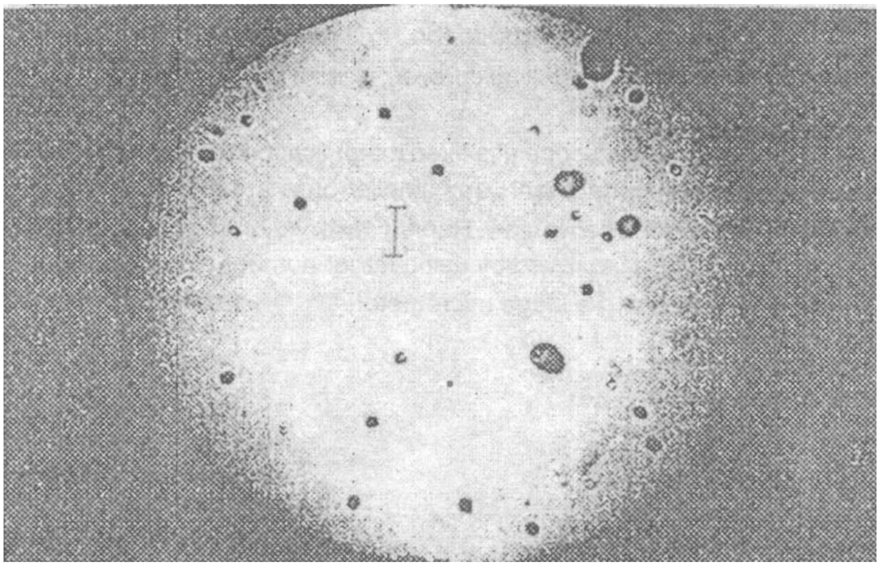


Fig. 3.15 A view of the alpha tracks in CR-39

Chapter 4

HEALTH PHYSICS AND RADIATION PROTECTION

Radiation safety/health physics personnel form an integral part of a radioactive laboratory. They ensure that all personnel working in the radiochemical laboratories follow good radiation safety/health physics practices. They also keep a vigil on the levels of radioactivity in the air as well as on the floors and working surfaces[1].

As such radioactivity is ubiquitous in nature. Uranium and thorium are radioactive, as are products in their decay chain except the last member. Uranium (^{238}U) and its daughter products together are called 4n+2 series or the uranium series and all the daughter products along with decay paths are shown in Fig. 4.1. The relevant nuclear data for this series are given in Table 4.1. Similarly decay paths and nuclear data for thorium (4n series) and ^{235}U (called actinium or 4n+3 series) are given in Fig. 4.2 and 4.3 respectively and Relevant nuclear data are given in Tables 4.2 and 4.3 respectively. In addition, a number of radioisotopes that exist in nature are given in Table 4.4

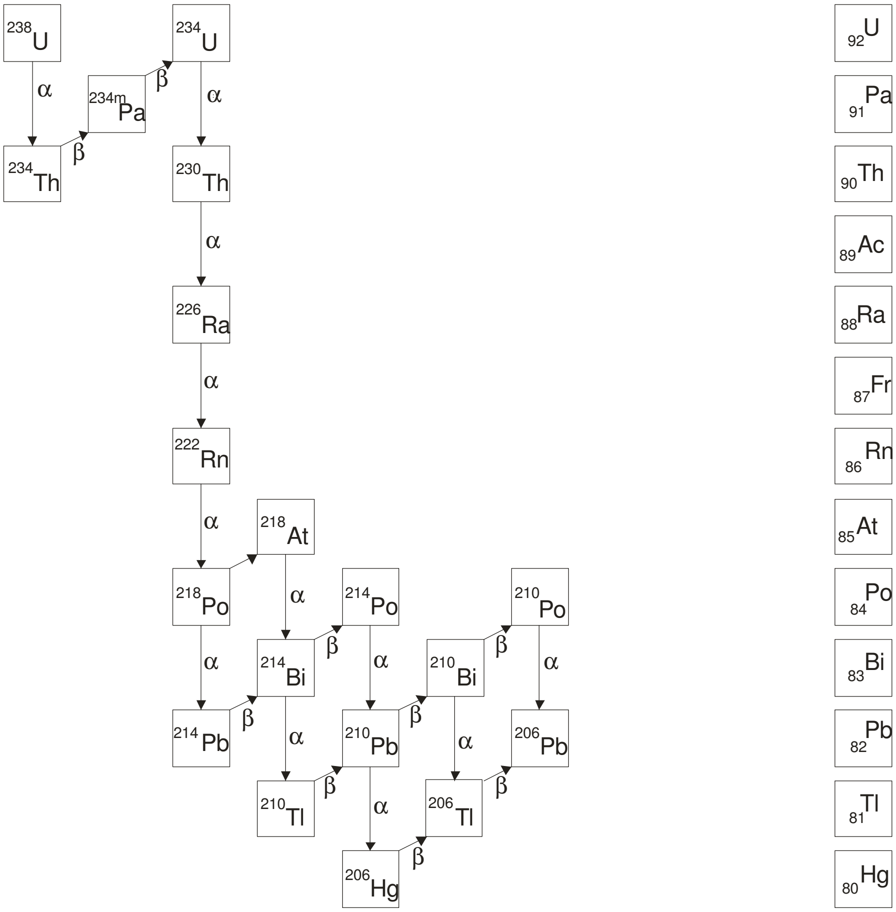


Fig 4.1 Uranium (4n + 2) Series

---

[1] Although ideally no radioactivity is expected to be present in air or on floor or on any other surface in a radiochemical laboratory, periodic measurements have to be made as a part of surveillance.

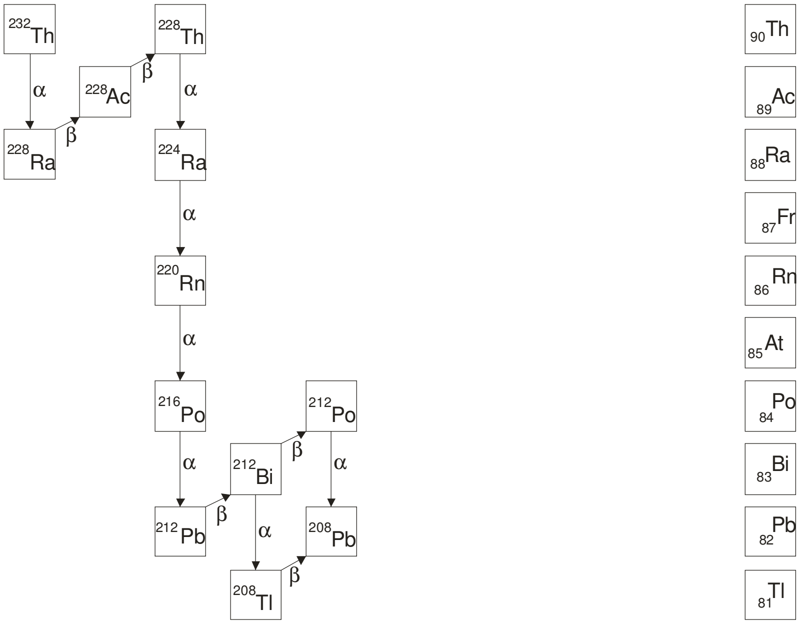


Fig 4.2 Thorium (4n) Series

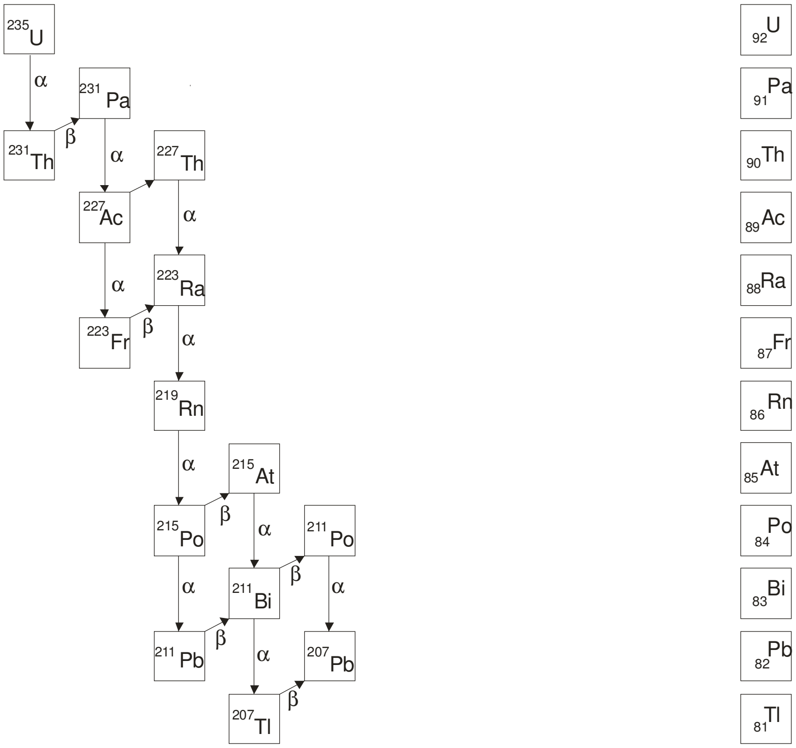


Fig 4.3 Actinium (4n + 3) series

Table 4.1 - Nuclear data for prominent members of ^{238}U series[2]

Isotope	Half Life	Type of decay	Energy, MeV
^{238}U			
^{234}U			
^{234m}Pa			
^{234}Pa			
^{234}Th			
^{230}Th			
^{226}Ra			
^{222}Rn			
^{218}Po			
^{214}Po			
^{210}Po			
^{214}Bi			
^{210}Bi			
^{214}Pb			
^{210}Pb			

Table 4.2 - Nuclear data for prominent members of natural thorium series

Isotope	Half Life	Type of decay	Energy, MeV

---

[2] When the nuclide emits radiations having more than one energy, energy corresponding to maximum intensity is given in the Table 1-4.

Table 4.3 - Nuclear data for prominent members of Actinium series

Isotope	Half Life	Type of decay	Energy, MeV
^{235}U			
^{231}Pa			
^{231}Th			
^{227}Th			
^{227}Ac			
^{223}Ra			
^{223}Fr			
^{219}Rn			
^{215}Po			
^{211}Po			
^{211}Bi			
^{211}Pb			
^{207}Tl			

Table 4.4 - Other naturally occurring radioisotopes

Radioisotope	Abundance	Half Life	Type of decay	Decay Product
^{40}K				
^{50}V				
^{87}Rb				
^{113}Cd				
^{115}Sn				
^{123}Te				
^{138}La				
^{144}Nd				
^{147}Sm				
^{148}Sm				
^{152}Gd				
^{176}Lu				
^{174}Hf				
^{187}Re				
^{190}Pt				

Alpha Radiation

Alpha particles lose energy rapidly while passing through a medium, because of their relatively high specific ionization, and can be stopped even by very thin absorbing materials. Thin sheet of a paper or thin (less than 0.3 mm) aluminium foil will absorb alphas from naturally α emitting sources. Alphas emitted by radioisotopes will travel only a few centimeters in air. The outer layer of skin (about 7 mg/cm^2) on our body will absorb α particles up to about 7.5 MeV. Since this is dead tissue, no harmful effect is produced upon the body. Therefore, α radiation is not considered as an external exposure hazard. However, if α-emitting radioisotopes enter human body, alpha radiation causes large internal exposure.

Beta Radiation

The processes by which β particles lose energy in absorbers are similar to those for alphas. However, β particle penetration in an absorber is much deeper than that of an α particle. This is due to its smaller mass, charge and higher velocity as compared to that of an alpha particle of similar energy. A 1 MeV β particle will travel about 3 m in air. β particles with energy above 70 keV penetrate the dead layer of the skin. Thus β radiation is an external radiation hazard.

The thickness and choice of material for shielding from β radiation depends upon: (1) ability to stop the most energetic β particles and (2) Bremsstrahlung radiation[3]. The shielding thickness, which is necessary to stop β particles of a given energy, will decrease with increasing density of shielding material. For example, 1.5 MeV betas can be stopped by about 0.025 cm thick aluminium ($\rho = 2.7$ g cm^{-2}) compared to 0.006 cm thick lead ($\rho = 11.3$ g cm^{-2}).

Gamma Radiation

The penetrating power of X-rays is well known and forms the basis for medical diagnostics and airport baggage inspection. The energy of X-rays in these devices is normally around 50 keV. Gamma rays, which like X-rays[4], are electromagnetic radiation, have energy ranging from a few keV to several MeV. High energy gamma rays, like those from ^{60}Co, are highly penetrating and are used to detect flaws and cracks in metallic components. High doses of gamma rays also kill body cells and this forms the basis of cancer treatment with ^{60}Co and ^{137}Cs gamma ray sources. Great care is, therefore, needed while working with gamma emitting nuclides. When gamma rays are incident upon a material, they may (i) pass through without any attenuation, (ii) get scattered by electrons in the medium (Compton Effect) or (iii) get absorbed (photoelectric and pair production effects). Low energy gamma rays are more readily attenuated. Similarly the attenuation is higher by high Z materials. Gamma radiation is virtually not attenuated by air and the radiation field can, therefore, be measured by instruments kept away from the source. Great care has to be taken to ensure that the radiation field in the work place is well below that stipulated by safety regulations. The radiation field can be reduced by interposing a shielding material between the source

---

[3] When electrons are accelerated in the electric field of a nucleus, continuous electromagnetic radiation is emitted and this is known as Bremsstrahlung radiation. The production of Bremsstrahlung increases with increasing atomic number of the absorber. A β source with $\beta_{max} = 1$ MeV will lose about 3%, of its energy as Bremsstrahlung when lead ($Z = 82$) is the absorber. If aluminium ($Z = 13$) is the absorber, the fraction is about 0.4%.

[4] Origin of X-rays and γ-rays are respectively due to atomic and nuclear phenomena.

and the personnel. The radiation field, to which a person is exposed, can also be brought down by increasing the distance between the source and the person as the field is inversely proportional to the square of the distance. The use of long tongs, or shielded cells with remote manipulators is, therefore, essential for handling radioactivity beyond a few milliCuries. Lead, steel and concrete are most commonly used radiation shields. The concept of half value thickness is quite useful for deciding shielding material. This is the thickness of a material required to reduce radiation intensity to half its original value. For example, half value thickness of lead for ^{137}Cs gamma rays (662 keV) is 0.55 cm. To reduce radiation field by a factor of 8, three half value thickness of lead (i.e. 1.65 cm) is required.

It is essential to follow good laboratory practices while working with radioactivity. Knowledge of radiation dose in the working environment is essential. This is measured on a routine basis using radiation dose meters. While personnel are leaving the laboratory, working place and hands of the personnel have to be monitored. Photographs of some health physics instruments are given in Figs. 4.4 to 4.6.

The experiments described in this Chapter are designed to measure natural radioactivity present in air and solid samples. Similar procedures are used for measuring radioactivity in samples from radiochemical laboratories.

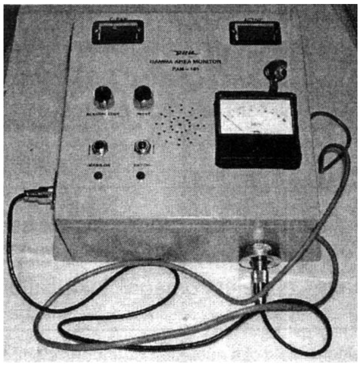


Fig. 4.4 Photography of a Gamma area monitor with a GM probe (Audio alarm and visual display are provided).

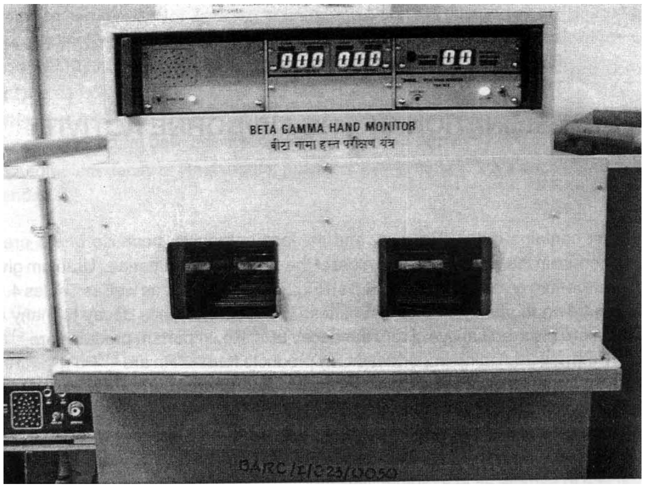


Fig. 4.5 Photograph of a hand contamination monitor.

Fig. 4.6 Photographs of different radiation monitors. (1. Teletector, 2. Alpha monitor, 3. Gun monitor, 4. Radiation survey meter, 5. Minirad survey meter, 6. Bicon survey meter, 7. Digi dose personal monitor and 8. TLD badge).

Experiment 12

DETERMINATION OF THE AIR BORNE ACTIVITY

Discussion

Ambient air contains radon (Rn) gas, and its decay products, because of the presence of uranium and thorium in the soil as well as in most of the construction materials. Radon isotopes are decay products of Uranium (^{222}Rn), ($t_{1/2}$= 3.8 d) and thorium (^{220}Rn ($t_{1/2}$= 55.6 s) respectively. Figures 4.1 and 4.2, as well as Tables 4.1 and 4.2 give details regarding thorium and uranium series. The radon isotopes decay to many daughter products, which are also radioactive. From the decay of ^{222}Rn, important products are ^{218}Po ($t_{1/2}$ = 3.1m) and ^{214}Pb ($t_{1/2}$ = 26.8 min). Important decay products from ^{220}Rn are ^{212}Pb ($t_{1/2}$ = 10.6 h) and ^{212}Bi ($t_{1/2}$ = 60.5 min). These decay products get accumulated on dust particles in air. If ambient air is sampled using high efficiency particulate air (HEPA) filters, then these aerosols are retained on the filter paper. Average alpha count rate in Mumbai area is around 5 dpm/m^3 of air. In a radioactive laboratory count rate might be more than this due to handling of radioactivity. It is a common practice to sample air in a radioactive laboratory by sucking it through a Glass Fiber Filter Paper (GFFP) for estimating air borne activity. The GFFP is loaded in a brass sampling head backed by a wire mesh. The other end of the sample head is attached to a suction pump with rubber tubing. An air flow of 40 litres/minute (double the breathing rate of humans) is maintained across the filter paper. The GFFP has a trapping efficiency of 99.98% for a particle size of 0.3 nm while the efficiency for all other particle sizes is higher. The activity attached to aerosols (dust particles) gets collected on the surface of the filter paper by impaction and interception.

Materials Required

A suction pump, brass sampling head, wire mesh, 2 meter long rubber tubing, glass fiber filter paper, counting set-up with ZnS(Ag) detector and GM detector.

Procedure

1. Load the filter paper of 33 mm dia in the air sampling assembly.
2. Put on the pump and suck the air through air sampler for 8 hours.
3. Towards the end of air sampling period, assemble the electronic units of the detectors and make connections as given in the instruction manuals. Note down the settings.
4. Switch on the power supply. After 10 minutes put on the 'EHT' of ZnS(Ag) counter and GM counter. Allow the system to stabilize for half an hour.
5. Obtain the background count rates for both counters for 600 s each.
6. Put off the pump and remove the filter paper from the air sampler for counting and note down time.
7. Pull out the sample rack of ZnS(Ag) counter and place the filter paper. Push the sample rack back completely such that the filter paper is inside the counter (since alpha ranges are small, it is essential to keep the sample as close to the detector face as possible).
8. Count the sample for 600 seconds and take out the filter paper.
9. Mount the sample in a groove of the GM counter stand. After 20 minutes delay to allow decay of ^{218}Po, count beta activity collected on the filter paper for 600 s.

NOTE: For complete decay of Rn daughter products, particularly ^{212}Pb a decay time of 72 hours is recommended.

Observations

Date

ZnS(Ag) Counter No. :
Source : Air sample
Function : Time
Preset time : 600 s
Paralysis time : 250 μs
Operating voltage : V
Position : Inside counter
Background count/60 s (C_b) :

Table 4.5 - Counting data of air sample using a ZnS(Ag) counter

Sl. No.	Counts 600 s	Counts/60 s C	Net counts/60s $C-C_b$
1			
2			
3			
4			
5			

GM Counter No. :
GM tube No. :
Function : Time
Preset time : 600 s
Paralysis time : 250 μs
Operating voltage : V
Position : Groove No.
Source : Air sample
Background counts/1800s:
Background count/60 s (C_b) :

Table 4.6 - Counting data of air sample using a GM counter

Sl. No.	Counts 600 s	Counts/60 s C	Net counts/60s $C-C_b$
1			
2			
3			
4			
5			

Calculations

A. α-activity assessment

Air Sampling duration : 8 h
Suction rate : 40 L/min
Total air sucked : $8 \times 60 \times 40 \times 10^{-3}$ m^3 = 19.2 m^3
Counting Efficiency : 33%
Let Count rate be : C_1 cpm
Activity on filter paper A_1 : [100 x C_1/33] dpm
Derived air concentration (DAC)[5] for Rn : [1.11×10^4] Bq m^{-3}=6.6×10^6 dpm m^{-3}

---

[5] Derived Air Concentration (DAC) is defined as the activity in Bq/m^3 of air for the isotope in question. DAC for an isotope is derived by dividing annual limit of intake (ALI) by the product of occupancy hours (2000 h) in an year and breathing rate (1.2 m^3/h or 2400 m^3 per year). So DAC = ALI/2400

Number of DAC of Rn : A_1/[6.6 x 10^6 x 19.2]
Air borne natural alpha radioactivity : DAC

B. β-activity assessment

Let Count rate be : C_2 cpm
Counting Efficiency : 12%
Activity on filter paper A_2 : [100 x C_2/12] dpm
Derived air concentration (DAC) for Pb : 740 Bq m^{-3} = 4.4 x 10^4 dpm m^{-3}
Number of DAC of Pb : A_2/[4.4 x 10^4 x 19.2]
Air borne natural beta radioactivity : DAC

Experiment 13

DETERMINATION OF RADIOACTIVITY IN SURFACE SOIL, CEMENT AND FLY ASH

Discussion

Uranium and thorium present in soil/rock contribute to the presence of radioactivity in a variety of matrices we come across in our daily life. Some processes lead to enhanced concentration of radioisotopes. For example, burning of coal leads to higher levels of radioactivity in coal fly ash (activity/unit weight) than in coal. In addition, coal fly ash enters the respiratory system easily and results in internal exposure. This experiment aims at providing first hand information on the levels of radioactivity in surface soil, cement and coal fly ash.

Materials required

NaI(Tl) counter set up, solid samples of surface soil, cement and coal fly ash (100 g each), sample holders (7.5 cm dia x 1 cm depth, with cover) - 8 nos., standard soil sample prepared by mixing 200 mg each of natural uranium and thorium nitrates in 9.6 g of surface soil, and a weighing balance.

Procedure

1. Assemble the electronic units of NaI(Tl) with single channel analyzer and make connections as given in the instruction manual. Note down the settings.
2. Switch on the mains supply. After 10 minutes put on the 'EHT'. Allow to stabilize for half an hour.
3. Calibrate the NaI(Tl) detector for energy using standards ^{137}Cs and ^{60}Co (as described in Experiment 4).
4. Adjust the single channel analyzer so as to measure γ-rays up to 2 MeV energy.
5. Keep an empty sample holder, along with the cover, touching the face of the detector and count background for 30 min. Calculate the background count rate per second (C_b).
6. Similarly obtain background count rate per second (C_b) for each sample holder.
7. Take one of the sample holders with cover and weigh it. Fill 10 g of standard soil sample in this sample holder and cap it.
8. Mount this sample touching the face of the detector. Note the counting time required to accumulate 10000 counts. Calculate counts per second per kg of material (C_S).
9. Take another sample holder. Weigh it and fill surface soil sample in this sample holder up to the brim and cap it. Weigh it and note the weight of the surface soil.
10. Mount the sample touching the face of the detector. Note the counting time required to accumulate 10000 counts. Calculate counts per second per kg of material (C_S).
11. Repeat steps 9 and 10 with the samples of coal fly ash and cement.

Observations

Date

NaI(Tl) Counter No. :

Function : Time Mode: Integral

Source : Standard soil sample, and environmental solid samples

Position : Sample touching the detector face to face

Table 4.7 - Counting Data for environmental samples.

Sl. No.	Sample	Wt. of sample, g	Counting time, s	Specific counts rate (cps/kg), C_S	Background, cps (C_b)*	Corrected (cps/kg) C_S-C_b	Mean Activity (Bq/kg)
1.	Standard Soil (10g)						
2.	Surface soil						
3.	Coal Fly Ash						
4.	Cement						

*Take background count rate for each empty holder before filling with sample.

Calculations

1. Background correction: Subtract the background count rate from the count rates of the samples and standard.
2. Total specific activity in the standard (A_s) = Bq (Calculated from weights of U and Th)
3. Specific count rate measured (C_s) =
4. Conversion factor (CF) = A_s/C_s =
5. Obtain absolute count rate of the environmental solid samples by multiplying the Corrected count rates with CF[6].

Comment

The same procedure can be used to measure the natural activity in milk powder. The activity in milk powder is due to presence of ^{40}K in it. (E_γ = 1460 keV, $t_{1/2} = 1.227 \times 10^9$ y)

---

[6] All the values you have obtained are based on the assumption that the ratio of U/Th ratio in the sample is same as that of the standard.

Chapter 5

RADIOACTIVITY AND RADIOCHEMICAL SEPARATIONS

A brief introduction to radiochemical and radioanalytical methods is given in Chapter 2, Section 1.4. As in conventional chemical separations, the chemistry of the elements forms the basis for any radiochemical separations. The main differences are that: (i) one deals with radioisotopes which emit ionizing radiation, (ii) amounts of radioelements are often in sub microgram range, (iii) some radioisotopes may be short lived and separation procedures have to be fast (minutes to seconds or even less) and (iv) the separated substance should have good chemical and radionuclide purity. In this chapter experiments involving radiochemical separations, preparation of the samples and the measurement of radioactivity using either a GM counter or a gamma ray spectrometer are described. These experiments are chosen so as to give an idea about radiochemical separation techniques, half life determination and radioactive equilibrium. Normally a filter chimney is used to separate the precipitate on a filter paper circle of 25 mm dia which can be directly used for counting. This ensures sample geometry and also a reproducible sample to detector geometry. A typical schematic of a filter chimney made out of Perspex is given in Fig 5.1.

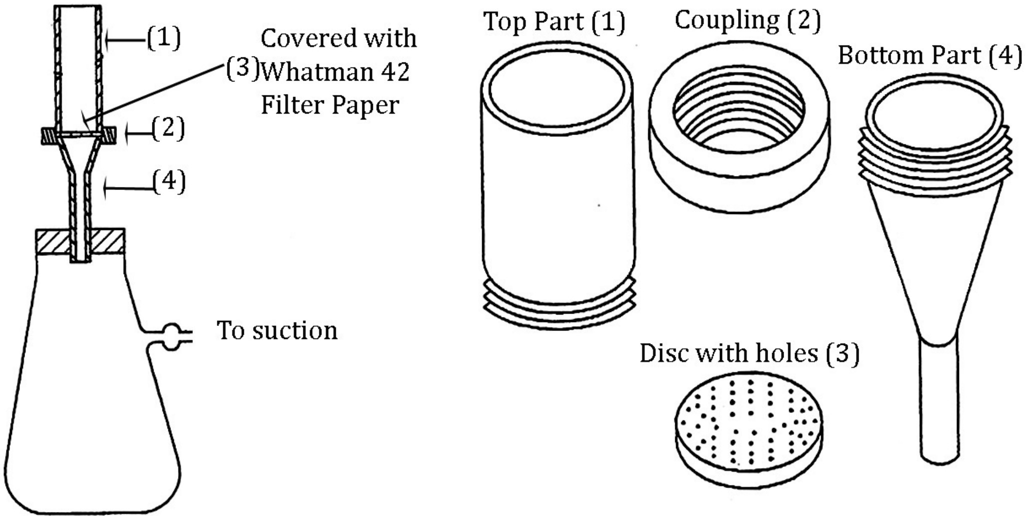


Fig. 5.1 Schematic of a filter chimney.

Experiment 14

HALF-LIFE DETERMINATION OF ^{137m}Ba

Discussion

Half-life is the characteristic of a radioactive nuclide. As discussed in Chapter 1, activity of a radioactive isotope at any time t is given by:

$$A = A_0e^{-\lambda t} \qquad \text{or} \qquad \ln A = \ln A_0 - \lambda t \tag{5.1}$$

where A and A_0 are the activities of the isotope at zero time and any time t respectively, and λ is the decay constant. Decay constant is equal to

$$\lambda = 0.693/t_{1/2} \tag{5.2}$$

where $t_{1/2}$ is the half-life of the radioactive isotope. Half-life is determined from the measured activity of a purified sample at different time intervals.

In this experiment, half-life determination of ^{137m}Ba is described. ^{137m}Ba is a short lived daughter product of ^{137}Cs and the decay chain is as follows:

$$^{137}Cs \xrightarrow[30y]{\beta^-} {}^{137m}Ba \xrightarrow[2.55\,\text{min}]{IT} {}^{137}Ba \tag{5.3}$$

As the half-life of ^{137m}Ba is very short compared to ^{137}Cs, it comes into secular equilibrium with ^{137}Cs within 10 min of purification of ^{137}Cs. In this experiment Ba has to be separated from its parent and for this a ^{137}Cs-Ba generator is prepared and used. A solution containing ^{137}Cs, in equilibrium with ^{137m}Ba, is taken in 1 N ammonium nitrate solution and loaded onto an ion exchange column containing ammonium molybdophosphate (AMP). Cesium and barium are retained on the ion exchanger. Barium is selectively eluted from the column with 0.1 N ammonium nitrate solution in 0.5 M HNO_3. Using a NaI(Tl) detector, radioactivity 'A' of ^{137m}Ba is measured as a function of time over a period of 30 minutes. A straight line will be obtained when ln A is plotted as a function of time. The slope of this line is equal to λ, from which half-life can be calculated.

Materials Required

Ammonium molybdophosphate, ammonium nitrate, nitric acid, distilled water, 5000 Bq of ^{137}Cs, glass column (5 mm dia. x 10 cm), standard flasks, transfer pipettes, syringe, beakers, counting tubes, asbestos powder, NaI(Tl) detector with accessories and semi log papers.

Procedure

1. Assemble NaI(Tl) detector setup. Make the connections as given in the instruction manual. Put on mains and apply high voltage in small steps until the rated voltage is reached.
2. Obtain γ-spectrum of ^{137}Cs by operating in differential mode. (See experiment 4, Chapter 3 for details).
3. Use this spectrum to fix the base line voltage and window for measuring 662 keV γ-rays. Keep an empty counting vial in the well and count for 30 min to obtain the background count rate.
4. Mix 2 g of AMP with 1 g of fine fibres of asbestos and soak this in 1 N ammonium nitrate solution to make a paste.

5. Transfer the paste into the glass ion exchange column up to a height of 2 cm. Pass 10 mL of 1N ammonium nitrate solution. Keep the flow rate of 10 drops/min.
6. Take ^{137}Cs activity in 1 N ammonium nitrate solution and transfer onto the column. Wash with 10 mL of 1 N ammonium nitrate solution. Collect the washings in a beaker.
7. Wash the column with 2 mL of 0.1 N ammonium nitrate (in 0.5 N HNO_3) to remove ^{137m}Ba if present. Collect the eluant in the same beaker and wait for 20 min.
8. Pass another 2 mL of the above solution through the column and collect the eluant in a counting tube.
9. Remove the counting tube and place in the well of NaI(Tl) detector (which is already kept on and calibrated).
10. Count the sample for 20 s[1] and record the count rate in the tabular form. Repeat this at 1 min intervals for 15 min.
11. Correct the count rates by subtracting background count rate (step 3). Plot the corrected count rates on Y-axis and time on X-axis of a semilog paper. The resulting plot would be a straight line (eqn. 5.1). Your plot will be like Fig. 5.2.
12. From the plot choose a convenient activity A_1 and its corresponding time t_1. Note the time t_2 corresponding to the activity $A_1/2$. $t_2 - t_1$ gives the half life of ^{137m}Ba.
13. Also obtain the slope of the straight line to get λ and calculate half-life.

Observations

Date

Detector	: NaI(l) well type	High voltage	: 700 V
Mode	: Differential	Amplifier Gain	:
Window	: V	Base line	: V
Function	: Time & Operate	Sources	: ^{137}Cs-Ba
Position	: In the detector well	Preset Time	: 20 s
Background counts/300s :		Background count/20 s (C_b)	:

Table 5.1 - Counting Data

Sl. No.	Time From Start	Counts/ 20 s	Counts $C_i - C_b$	Sl. No.	Time From Start	Counts/ 20 s	Counts $C_i - C_b$
1				9			
2				10			
3				11			
4				12			
5				13			
6				14			
7				15			
8				16			

---

[1] Since the half-life of ^{137m}Ba is short (2.55 min) and counting time is comparable (20 s), a good fraction decays during the counting period. This might lead to a slight error in the determined half-life.

Calculations

1. *Half life from step 12*

Al = counts/20s t_1 = min

$A_{1/2}$ = counts/20s t_2 = min

Half life = t_2-t_1 = min

2. *Half life from Step 13*

Slope = $\Delta(\log A)/\Delta t = (\log A_1 - \log A2)/(t_2-t_1)$ = min^{-1}

Half life = 0.693/slope x 2.303 = min.

3. *Half life from eqn. (5.1)*

Extrapolate the graph to obtain activity at zero time A_0.
Then obtain activity A when t is 5 min =
Calculate by substituting these values in eqns. (5.1)

λ = min^{-1} $t_{1/2}$ = min

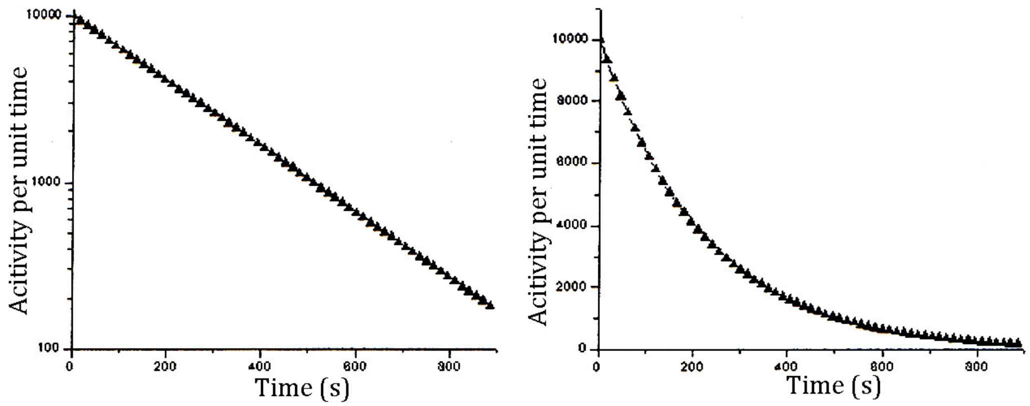


Fig. 5.2 Radioactivity of ^{137m}Ba as a function of time.

Conclusions and Suggestions

1. Write your conclusions.
2. Plan the experiment to determine the half life of ^{212}Bi obtained from step 11, Experiment 16 in this Chapter.
3. Plan an experiment to measure the growth of ^{137m}Ba as follows:
   (a) Wash the column with 2 mL of 0.1 M ammonium nitrate in 0.5 M HNO_3. Reject the eluant.
   (b) After 2 min, wash the column with 2 mL of the above solution and collect the eluant into a counting tube. Count it for 20 s using NaI(Tl) system.
   (c) Repeat step (b) after waiting for growth of ^{137m}Ba for 4 min, 6 min, 10 min, 15 min and 60 min.
   (d) Plot the activity on Y-axis and time on X-axis on a semi log paper to obtain the growth curve of ^{137m}Ba. (Your plot should look like curve d of Fig. 5.3)
   (e) Calculate the times corresponding to 50%, 75% and 87.5% growth which should be equal to 1, 2 and 3 half lives of ^{137m}Ba.

2. RADIOACTIVE EQUILIBRIUM

In the case of genetically related radionuclides, like those of the naturally occurring radioactive series of ^{238}U and ^{232}Th, both parent and daughter nuclides contribute to radioactivity. When a radioactive nuclide decays to its daughter product, which is also radioactive, the rate of formation of the daughter is given by:

$$\frac{dN_2}{dt} = N_1\lambda_1 - N_2\lambda_2 \qquad (5.5)$$

where N_1 and N_2 are the atoms of parent and daughter nuclides present at any time t, and λ_1 and λ_2 are decay constants of parent and daughter respectively. Daughter activity ($A_2 = N_2 \lambda_2$) at any time t is obtained by solving differential eqn. (5.5)

$$A_2 = A_1^0 \frac{\lambda_2}{\lambda_2 - \lambda_1}(e^{-\lambda_1 t} - e^{-\lambda_2 t}) + A_2^0\, e^{-\lambda_2 t} \qquad (5.6)$$

where A_1^0 and A_2^0 are the initial activities of parent and daughter nuclides respectively. The second term in eqn. (5.6) is the initial amount of daughter product present at time t. In the case of freshly purified parent, at t = 0 no daughter atoms are present ($N_2^0 = 0$). Daughter activity is only due to daughter formed from parent's decay, which is given by the first term in eqn. (5.6). The second term is equal to zero. When $\lambda_1 < \lambda_2$ (parent is longer lived) and for sufficiently large values of t, an equilibrium is reached between the activity of parent and daughter. Eqn. (5.6) is then reduced to the form:

$$A_2 = A_1^0 \frac{\lambda_2}{\lambda_2 - \lambda_1} e^{-\lambda_1 t} \qquad (5.7)$$

since $A_1^0\, e^{-\lambda_1 t}$ is equal to A_1, eqn. (5.7) can be written as:

$$A_2 = A_1 \frac{\lambda_2}{\lambda_2 - \lambda_1} \text{ or } \frac{A_2}{A_1} = \frac{\lambda_2}{\lambda_2 - \lambda_1} \qquad (5.8)$$

which means that the daughter activity will be greater than the parent activity by a constant factor $\lambda_2/\lambda_2-\lambda_1$. This steady state is known as radioactive equilibrium.

When the ratio of λ_1 and λ_2 is approximately equal to 0.1, then the equilibrium is called transient equilibrium. An example of transient equilibrium is given below.

$$^{212}\text{Pb} \xrightarrow[10.64\text{h}]{\beta^-} {}^{212}\text{Bi} \xrightarrow[1.01\text{h}]{\beta^-} {}^{212}\text{Po} \qquad (5.9)$$

A typical activity profile for this system is given in Fig. 5.3.

When $\lambda_1 << \lambda_2$ (parent is much longer lived than the daughter) eqn. (5.8) is reduced to:

$$A_2 = A_1 \qquad (5.10)$$

This condition when the daughter activity is equal to parent activity is known as secular equilibrium. An example is ^{144}Ce decay and is given below:

$$^{144}\text{Ce} \xrightarrow[285\text{d}]{\beta^-} {}^{144}\text{Pr} \xrightarrow[17\text{m}]{\beta^-} {}^{144}\text{Nd (Stable)} \qquad (5.11)$$

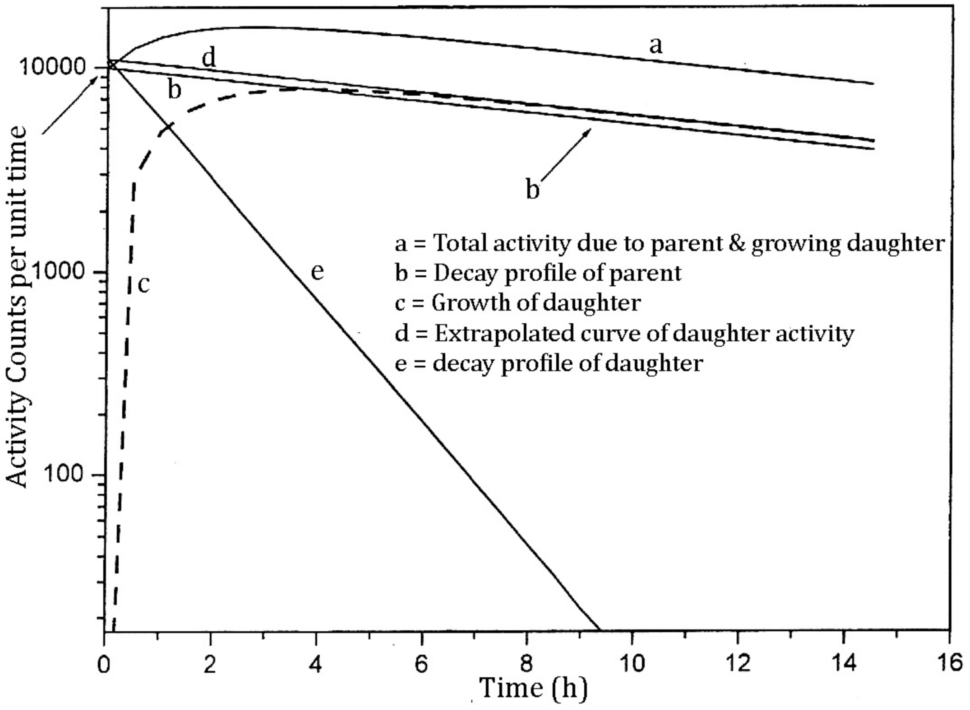


Fig. 5.3 An example of transient equilibrium. Decay profile of ^{212}Pb-^{212}Bi

Total activity A which the sum of activities due to the purified parent (A_1) and growing daughter (A_2) at any time t after purification is given by:

$$A = A_1 + A_2 = A_1^0 e^{-\lambda_1 t} + A_1^0 \frac{\lambda_2}{\lambda_2 - \lambda_1}(e^{-\lambda_1 t} - e^{-\lambda_2 t}) \qquad (5.12)$$

After equilibrium, total activity decreases with the decay constant of the parent as given by equation (5.13)

$$A = A_1^0 \, e^{-\lambda_1 t} + A_1^0 \frac{\lambda_2}{\lambda_2 - \lambda_1} \, e^{-\lambda_1 t}$$

$$A = A_1^0 \left[1 + \frac{\lambda_2}{\lambda_2 - \lambda_1} \right] e^{-\lambda_1 t} \qquad (5.13)$$

In the case of secular equilibrium, total activity is given by:

$$A = 2 \, A_1^0 \, e^{-\lambda_1 t} \qquad (5.14)$$

A typical activity profile of such a system is given in Fig 5.4

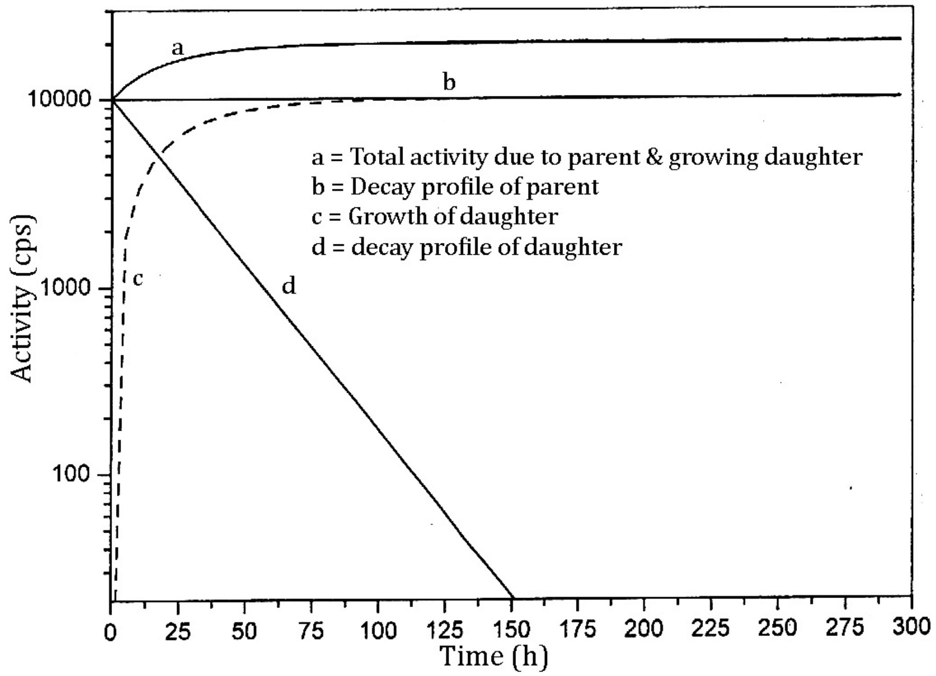


Fig. 5.4 An example of Secular Equilibrium. Decay profile of ^{144}*Ce-* ^{144}*Pr*

Experiment 15

SECULAR EQUILIBRIUM

Discussion

The aim of this experiment is to study the secular equilibrium and to determine half-life of the daughter product. ^{144}Ce -^{144}Pr (eqn. 5.11) is a typical example of secular equilibrium and is used in this experiment. ^{144}Ce is purified from its daughter ^{144}Pr by precipitating as ceric iodate. The time of separation is noted and the total activity of parent and growing daughter in the precipitate is measured at frequent intervals. The growth curve is analyzed to get the half-life of ^{144}Pr.

Materials Required

^{144}Ce, HNO_3, $KBrO_3$, HIO_3, Ce and La carriers having 10 mg of metal ion/mL, alcohol, aluminium plate or Perspex plate, double scotch tape, cellophane paper, filter paper circles, burner, filter chimney, G.M. counting set up with accessories and a sample stand.

Procedure

1. Assemble the GM counter set up and make connections as given in the instruction manual. Note down the settings.
2. Switch on the mains supply. After 10 minutes put on the 'EHT'. Allow to stabilize for half an hour.
3. Take the solution containing ^{144}Ce-^{144}Pr activity in a centrifuge tube and add 20 mg each of Ce^{3+} and La^{3+} carriers and 8 mL of conc. HNO_3. Mix well and add about 0.5 g of $KBrO_3$. Heat the solution to oxidise Ce^{3+} to Ce^{4+} and add 20 mL of 0.3 M HIO_3. Ensure complete precipitation. Note down the clock time and it is zero time. Cool, centrifuge and filter the precipitate of $Ce(IO_3)_4$ through a Whatman 42 filter paper using a filter chimney (Fig. 5.1)
4. Wash the precipitate with 20 mL of water followed by 10 mL of alcohol.
5. Remove the filter paper from the filter chimney and mount on an aluminium or Perspex plate and cover the precipitate with cellophane paper.
6. Measure the activity using a GM Counter initially at intervals of 2 minutes. Record the clock time before starting each counting. After the count rate becomes nearly constant, take readings once in half an hour. Tabulate the data.
7. Plot the activity of the sample on a semi log paper as a function of time. Take time on X-axis and activity on Y-axis. The plot should be similar to curve in Fig. 5.4.

Observations

Date
GM Counter No. : GM tube No. :
Operating voltage : V Paralysis time : 250 µs
Source : ^{144}Ce precipitate Position : Groove No.
Preset time : 60 s Background/60 s (Cb):
Zero time :

Table 5.2 - Counting data of the purified sample (^{144}Ce-^{144}Pr)

Sl. No.	Start Time hh-mm	Elapsed Time (min)	Observed Counts Per 60 s (C_0)	Corrected Counts $C = C_0 - C_b$
1.				
2.				
3.				
4.				
5.				
6.				
7.				
8.				
9.				
10.				
11.				
12.				
13.				
14.				
15.				
16.				
17.				
18.				
19.				
20.				
21.				
22.				
23.				
24.				

Calculations

1. Plot corrected count rate C on (Y axis) Vs. elapsed time t since precipitation on (X-axis) on a semi log paper (curve a). This plot gives activity of ^{144}Ce + ^{144} Pr as a function of time.
2. Draw a parallel line to the latter part of 'curve a' from zero time of 'curve a' to obtain 'curve b'. This represents activity due to ^{144}Ce alone. Since ^{144}Ce is long lived, the decrease of its activity in the duration of the experiment is negligible. Curve b is, therefore, nearly parallel to the X-axis.

3. Obtain growth curve of ^{144}Pr activity by plotting the difference of activity (curve a- curve b) as a function of time (curve c).
4. Plot activity (curve b- curve c) as a function of time (curve d). This curve represents the decay of ^{144}Pr and is used to calculate half life of ^{144}Pr. Slope of this curve gives $\lambda_{^{144}\mathrm{Pr}} =$
5. Half life of ^{144}Pr :
   Your plot would be like Fig. 5.4.

Experiment 16

TRANSIENT EQUILIBRIUM

Discussion

Objective of this experiment is to demonstrate the growth and decay of activity in genetically related radionuclides and to determine the half-lives of parent and daughter products. ^{212}Pb and ^{212}Bi are the members of the thorium series (see Fig. 4.2). ^{212}Bi is the daughter product of ^{212}Pb (eqn. 5.9) and this pair of nuclides is an example for transient equilibrium. ^{212}Pb and ^{212}Bi are extracted by dithiozone in CCl_4 from a solution of natural thorium nitrate. ^{212}Pb is separated from ^{212}Bi by precipitating as lead sulphide. Time of separation is noted. Total activity in the precipitate, due to the decay of parent ^{212}Pb and growth of ^{212}Bi, is measured at frequent intervals. Growth and decay curves are analyzed to obtain half-lives of ^{212}Pb and ^{212}Bi.

Materials Required

G.M. counter with accessories, Kipp's apparatus, balance, filter chimney unit, 100 mL separating funnels, beakers, measuring cylinders, thorium nitrate, bismuth and lead carriers, tartaric acid, citric acid, ammonia, phenolphthalein indicator, HCl and 0.2% dithiozone in CCl_4.

Procedure

1. Assemble GM counter set up and make connections as given in the instruction manual. Note down the settings.
2. Switch on the mains supply. After 10 minutes, put on the 'EHT'. Allow to stabilize for half an hour.
3. Take 2 g of thorium nitrate and dissolve in about 20 mL of distilled water. Add 5 mg of Pb and 5 mg of Bi carriers. To this add 5 g of tartaric acid and 2 g of citric acid.
4. Add a drop of phenolphthalein and adjust pH to 9.5 by addition of ammonia (to a deep pink colour).
5. Transfer the solution to a 100 mL separating funnel and add 20 mL of 0.2% dithiozone in CCl_4. Shake vigorously.
6. Separate the organic and aqueous phases.
7. Repeat the extraction till the organic layer no longer turns deep pink and remains green.
8. Combine the organic phases and wash with aqueous ammonia (pH = 9.5). Discard the aqueous layer.
9. To the organic phase add HCl solution (pH = 3) and shake well to back extract lead. Separate the organic and aqueous layers. Repeat the back extraction with HCl (pH=3) until organic layer turns purple. Note the time of extraction (t = 0.). Combine the aqueous phases. This contains ^{212}Pb fraction (L).
10. To the organic fraction, add about 25 mL of 1 N HCl, shake well and separate the organic and aqueous layers. This aqueous phase contains ^{212}Bi fraction (B). Reject the green organic phase.
11. To fraction L, add about 10 mg of Pb carrier, heat to boiling and precipitate Pb as PbS by passing H_2S. After cooling, filter and dry the precipitate under infrared lamp. Mount the precipitate on an aluminium plate.
12. Measure the activity of the sample with a GM counter at intervals of 15 minutes over a period of 4 hours and afterwards at intervals of 2 hours over a period of 24 hours. Note down clock time of the start of counting in each case.

Observations

Date

GM Counter No. :
GM tube No. :
Operating voltage : V
Paralysis Time : 250 μs
Source : ^{212}Pb precipitate
Position : Groove No.
Preset time : 60 s
Background/60(C_b):
Zero time :

Table 5.3 - Counting data of the purified sample (^{212}Pb-^{212}Bi)

Sl. No.	Start Time hh-mm	Elapsed Time (min)	Observed Counts Per 60 s (C_0)	Corrected Counts $C = C_0 - C_b$
1.				
2.				
3.				
4.				
5.				
6.				
7.				
8.				
9.				
10.				
11.				
12.				
13.				
14.				
15.				
16.				
17.				
18.				
19.				
20.				
21.				
22.				
23.				
24.				

Calculations

1. Plot corrected count rate C on (X-axis) Vs. elapsed time t on (Y-axis) starting with t=0 (step 9) on a semi log paper (curve a). This plot gives activity of ^{212}Pb +^{212} Bi as a function of time.
2. Extrapolate this curve to zero time (Curve a). Draw a parallel line from A_0 to the later part of the curve a as shown in Fig. 5.3. (curve b). This curve represents the decay of ^{212}Pb.

3. Obtain growth curve of ^{212}Bi activity (curve c) by plotting the difference of activity (curve a - curve b) as a function of time.
4. Extrapolate curve c to zero time as shown in Fig. 5.3 to obtain curve d.
5. Plot activity (curve d - curve c) as a function of time (curve e). This represents decay of freshly isolated ^{212}Bi.
   (a) Slope of b = λ for ^{212}Pb =
   (b) Slope of e = λ for ^{212}Bi =
   (c) Half life of ^{212}Bi : (from slope of e) =
   (d) Half life of ^{212}Pb : (from slope of b) =

Comments

1. Activity of ^{212}Bi (curve c) is higher than the equilibrium activity of ^{212}Pb at equilibrium time. Determine ratio of activity of ^{212}Bi and ^{212}Pb, and check whether it is equal to

$$\frac{\lambda_{^{212}Bi}}{\lambda_{^{212}Bi} - \lambda_{^{212}Pb}} .$$

2. Add 10 mg of Bi carrier to fraction B from step 10. Precipitate Bi as sulphide. Filter the precipitate, dry and count as a function of time to determine the half life of ^{212}Bi.

This can be a demonstration experiment for M.Sc. students. ^{212}Bi activity can be milked from natural thorium salts.

3. FISSION YIELD DETERMINATION

Isotopes of heavy elements like thorium and uranium undergo division into two nearly equal parts (nuclear fission) when bombarded with neutrons. The isotope ^{235}U undergoes fission with very low energy (- 0.025 eV) neutrons and is called a fissile nuclide. In this case, the absorption of neutron leads to the formation of ^{236}U compound nucleus. Division of ^{236}U nucleus into two nearly equal parts is one of the prominent modes of de-excitation of this unstable nucleus. Release of energy (200 MeV per nucleus) and neutrons (2-3 per reaction) has made nuclear energy a reality, which today contributes about 16% of the total electricity in the world. The division of ^{236}U nucleus is not unique. It leads to the formation of a very large number of fission products with varying yields. Determination of mass yields forms an important aspect in experimental investigations of nuclear fission. A plot of variation of mass yield as a function of fission product mass is called mass distribution curve. Experimental mass distribution curves for the thermal neutron induced fission of ^{233}U, ^{235}U, ^{239}Pu and a mixture of 65% U + 35% Pu and are given in Fig. 5.5.

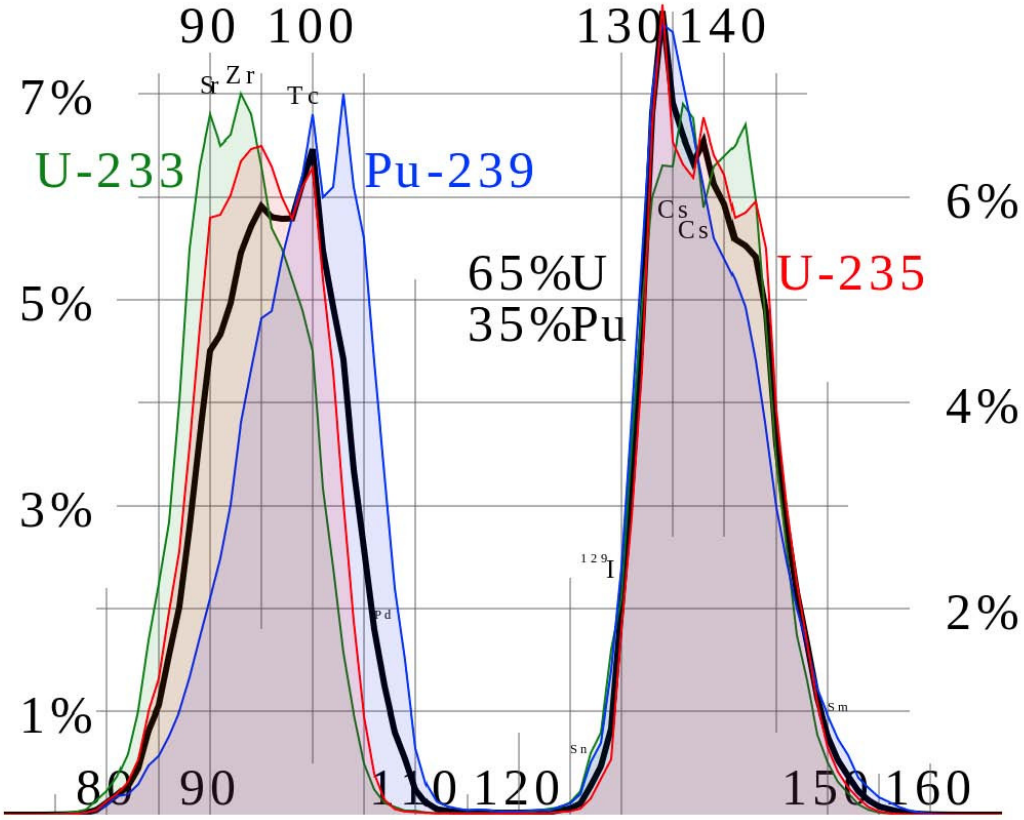


Fig. 5.5 Mass distribution curves in the thermal neutron induced fissions of ^{233}U, ^{235}U, ^{239}Pu and a mixture of 65% U + 35% Pu.

Activity A of a member of an isobaric chain in the nuclear fission is given by

$$A = N\sigma\varphi(1-e^{-\lambda t})e^{-\lambda T} \qquad (5.15)$$

where N is the number of atoms in the target, σ is the fission cross section (cm^2), φ is the neutron flux (n cm^{-2} s^{-1}), Y is the yield of the isobaric chain that has to be determined, λ is the decay constant and, t and T are the irradiation and cooling periods respectively. A is obtained from the measured count rate (C) at the time of separation, from the experiment, as:

$$C = A\ \varepsilon\ ChY \tag{5.16}$$

where ε is the efficiency of the detector and ChY is the chemical yield. From eqns. (5.15) and (5.16), fission yield, Y can be calculated as:

$$Y = C\frac{e^{\lambda T}}{\varepsilon\ ChY\ N\ \sigma\ \varphi\ (1- e^{-\lambda t})} \tag{5.17}$$

Experiment 17

FISSION YIELD DETERMINATION OF ^{91}Sr and ^{89}Sr

Discussion

Strontium isotopes ^{89}Sr, ^{90}Sr and ^{91}Sr are formed in the fission of isotopes like ^{235}U and ^{239}Pu. Strontium is separated from the fissioning element and other fission products by precipitating as nitrate using cold, conc. HNO_3. It is purified by $Fe(OH)_3$ scavenging. Finally, Sr is precipitated as carbonate and mounted on a suitable sample holder for counting. The activity data are used for fission yield determination. Since ^{90}Sr is long lived ($t_{1/2}$ = 28.5 y) as compared to ^{89}Sr ($t_{1/2}$ = 50.5 d) and ^{91}Sr ($t_{1/2}$ = 9.5 h), its contribution to activity in the sample is small.

Materials Required

Sr carrier: strontium chloride dissolved in water to obtain 10 mg of Sr/mL. Ba carrier: 10 mg of Ba/mL as barium chloride dissolved in water, Fe carrier: 10 mg Fe/mL as ferric chloride in very dilute HCl, HNO_3, CH_3COOH, ammonium acetate, Na_2CO_3, K_2CrO_4, NH_4OH, ice, pH paper, alcohol, GM counter set up, mounting plates, filter discs, filter chimney unit, oven and balance.

A stock solution containing mixed fission products (f.p.) is prepared by irradiating a few mg of uranium trioxide in a reactor for an hour and dissolving it in a known volume of HCl.

Procedure

1. Assemble the GM counter set up and make connections as given in the instruction manual. Note down the settings.
2. Switch on the power supply. After 10 minutes put on the `EHT'. Allow to stabilize for half an hour.
3. To an aliquot of the fission product solution add 2 mL each of Ba and Sr carrier solutions. Remove Cl^- ion by boiling with conc. HNO_3. Reduce the volume almost to dryness.
4. Add 20 mL of conc. HNO_3. Cool in ice for 5 to 10 min to let $Sr(NO_3)_2$ to crystallize. Centrifuge and discard the supernatant.
5. Dissolve the crystals in 10 mL of distilled water. Add 5-10 drops of Fe carrier and dilute NH_4OH and stir well. Centrifuge and discard the $Fe(OH)_3$ precipitate.
6. Neutralize the supernatant with 6 M HNO_3. Add 1 mL of 6 M acetic acid and 2 mL of 6 M ammonium acetate. Adjust the pH to 5. Heat the solution to boiling and add 2 mL of 2 M K_2CrO_4 solution while stirring. Continue heating for one minute. Allow the solution to cool and centrifuge. Discard the precipitate.
7. To the supernatant add slowly 2 M Na_2CO_3 solution. Heat to boiling. Allow to cool, centrifuge the solution and discard the supernatant. Note down clock time. Difference between the end of irradiation time and this time-gives cooling time (T) in eqn. (5.15).
8. Dissolve the precipitate in 1 mL of 6 M HNO_3. Dilute to 10 mL. Add 0.5 mL of Fe carrier. Repeat steps 5, 6 and 7.
9. Wash the strontium carbonate precipitate with water. Dissolve in 1 mL of dil. HNO_3. Add 0.5 mL of Fe carrier and then add slight excess of NH_4OH. Centrifuge and filter the supernatant through a Whatman 41 filter paper.
10. Add a slight excess of 2 M Na_2CO_3 solution to the supernatant, boil, cool and centrifuge. Discard the supernatant.

11. Wash the precipitate with distilled water, filter through a pre-weighed Whatman 41 filter paper, wash with alcohol and dry at 110°C for 10 min. Cool for 10 min, weigh and mount on an aluminium plate.
12. Measure the activity of the sample as a function of time.
13. Measure activity once in an hour for first 24 hours and later once in a week for two months. Note down time (T_3) before each counting.

Observations

Date

GM Counter No.	:	GM tube No.	:
Settings			
Operating voltage	: V	Paralysis time	: 250 µs
Source	: $^{89}Sr + ^{91}Sr$ as $SrCO_3$	Position	: Groove No.
Preset time	: 60s	Background/60 s (Cb):	
Irradiation time	:1h		
End of irradiation (hh-mm)	: (T_1)	Separation time (Step 7) (hh-mm)	: (T_2)
$T = T_2-T_1$	:	Elapsed time	: T_3-T2 for each reading

Table 5.4 - Beta activity counting data

Sl. No.	Start Time (T_3) hh-mm	Elapsed Time (min)	Observed Counts Per 60 s (C_0)	Corrected Counts $C = C_0 - C_b$
1.				
2.				
3.				
4.				
5.				
6.				
7.				
8.				
9.				
10.				
11.				
12.				
13.				
14.				
15.				
16.				
17.				
18.				
19.				
20.				
21.				
22.				
23.				
24.				

Wt. of Sr Carrier added (W_1) = mg   Wt. of Filter Paper (W_f) = mg

Wt. of Filter Paper + $SrCO_3$ = mg   Wt. of $SrCO_3$ ppt. (W_2) = mg

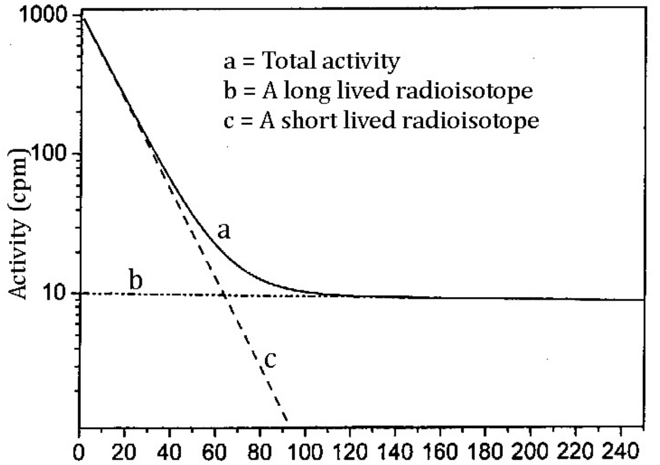
Wt. of Sr in $SrCO_3$ ppt. (W_3) = mg   Chemical yield (W_3/W_1) =

Calculations

1. Plot on a semi log paper the activity as a function of elapsed time taking time on X-axis and activity on Y-axis (curve a, Fig. 5.6).
2. From the later points (after 5 days) extrapolate the curve to zero time (curve b). Slope of curve b corresponds to λ of ^{89}Sr ($t_{1/2}$ = 50.5 d). Note the activity of ^{89}Sr corresponding to zero time. Plot the difference (curve a - curve b) as a function of time to obtain the decay curve of ^{91}Sr ($t_{1/2}$ = 9.5 h) (curve c). Note down the activity of ^{91}Sr at zero time. Your plot should look Similar to Fig. 5.6.
3. Calculate fission yields of mass chains 89 and 91 using eqn (5.17) and the zero time activity obtained in step 2.

Comments and Suggestions

1. It is to be noted that the activity of ^{90}Sr is not considered due to its long half-life ($t_{1/2}$ = 28.5 y).
2. Fission yields of mass chains of 89 and 91 in thermal neutron fission of ^{235}U are respectively 4.8% and 5.93%.



Activity profile of a mixture of long lived and short lived radioisotopes (not genetically related)

Fig. 5.6 Activity profile of a mixture of two radioisotopes that are not genetically related.

Experiment 18

FISSION YIELD DETERMINATION OF IODINE ISOTOPES

Discussion

Many iodine isotopes are formed in the fission of nuclides such as ^{235}U and ^{239}Pu. Prominent among them are $^{131\text{-}135}$I with half lives ranging from 53 min to 8 d. Fission product iodine is separated from the rest of the fission products by extracting as molecular iodine into CCl_4. Iodine products are formed in different oxidation states. A redox reagent like H_2O_2 or HNO_2 is used to convert all the products to molecular iodine. Then it is extracted into $CC1_4$ and back extracted into sodium bisulphite solution. The aqueous liquid sample is assayed for gamma activity using a high resolution gamma spectrometer consisting of HPGe detector and a PC based multi channel analyser (MCA). The activity data are used for fission yield determination.

Materials Required

Carrier solutions of iodine and tellurium (10 mg/mL), saturated sodium bisulphite solution, Conc. HNO_3, NaOH pellets, CCl_4, H_20_2, 100 mL separating funnels, 20mL standard counting vials, graduated pipettes, graph sheets, HPGe detector with accessories and PC based MCA.

A stock solution containing mixed fission products is prepared by irradiating a few mg of uranium trioxide in a reactor for an hour alongwith an Al catcher foil to absorb fission products and dissolving catcher foil in a known volume of NaOH solution.

Procedure

1. Assemble the HPGe detector set up and MCA, and make connections as given in the instruction manual. Note down the settings.
2. Ensure that the Dewar of HPGe is filled with liquid nitrogen well in advance. (> 6 hours).
3. Switch on the power supply. After 20 minutes, start applying high voltage at the rate of 100V/min until the rated voltage is reached. Allow to stabilize for half an hour.
4. To an aliquot of the fission product solution add 2 mL each of I^- and Te carrier solutions.
5. Transfer this into a 100 mL separating funnel. Add 10 mL of CCl_4, a few drops of conc. HNO_3 and a few drops of H_2O_2. Shake well and allow the aqueous and organic phases to separate. Organic layer will be of violet colour. Note down the clock time as separation time.
6. Transfer the organic phase to another separating funnel. Wash with 5 mL of dil HNO_3 and reject the aqueous phase.
7. Add to this 6 mL of saturated sodium bisulphite solution. Shake well. Disappearance of violet colour is the indication of stripping of iodine isotopes from organic phase. Allow both the phases to settle.
8. Transfer the aqueous phase (5 mL) to a counting vial for radioactive assay.
9. Place the vial in a sample holder using a marked shelf of the detector where absolute detection efficiency is already determined. Measure the activity of the sample as a function of time.
10. Measure activity once in an hour for the first 24 hours and later once in a day for two weeks. Note down clock time (T3) for each counting.

Observations

Date
Detector : HPGe
High voltage :
MCA : PHA mode
Calibration standard : ^{152}Eu
Shelf number :
Weight of I carrier : mg
Irradiation time : 1 h
End of irradiation (hh-mm) : (T1)
$T=T_2-T_1$:

Amplifier Model :
Coarse gain :
Fine gain :
Shaping time :
Sample : 5 mL liquid
Weight of Te carrier :
Separation time (Step 7) (hh-mm) : (T2)
Elapsed time : T_3-T_2 for each counting

Table 5.6 - Relevant nuclear data and the measured count rates for $^{131\text{-}135}$I isotopes

Isotope	Half life	γ energy (keV)	abundance	Mass Yield %	dps at the Separtation time	Relative Fission Yield (R)	Reported R
^{131}I	8.045 d	364	82.4	1.53			
^{132}I	2.28 h	667	101.3	2.01			
		772	78.0	2.01			
^{133}I	20.3 h	529	89.0	3.13			
^{134}I	53 min	847	96.0	4.03			
^{135}I	6.61 h	1260	34.9	4.09			

Table 5.7 - Counting data for Iodine Isotopes

Sl. No.	Start Time hh-mm	Elapsed time	Peak Areas					
			364 keV	529 keV	667 keV	772 keV	847 keV	1260 keV
1								
2								
3								
4								
5								
6								
7								
8								
9								
10								
11								
12								
13								
14								
15								
16								
17								
18								
19								
20								
21								
22								
23								
24								
25								

Calculations

1. Calculate the activity corresponding to separation time for each iodine isotope. Enter them in Table 5.6
2. Calculate fission yields using eqn.(5.17) and taking the value of ChY as 1.
3. Calculate the relative yields w.r.t. the yield of $^{135}1$ (Table 5.6)
4. Obtain the yield ratio from literature yields (Table 5.6) and compare your values.

Comments and Suggestions

1. Literature data on yields of $^{131\text{-}135}I$ are cumulative yields for mass chains 131-135, where as the values determined in this experiment are not.
2. Determine the half life of ^{134}I by plotting the peak area of 847 keV as a function of time. Check your value with literature value (Table 5.6).
3. Chemical yields can be determined by precipitating iodine as PbI_2 from aqueous medium and comparing the weight of iodine in the precipitate with the carrier weight.

4. SEPARATION OF RADIOISOTOPES FROM NATURAL URANIUM AND THORIUM

Experiment 19

SEPARATION OF ^{234}Th FROM NATURAL URANIUM USING ION EXCHANGE PROCEDURE

Discussion

Natural uranium contains 99.3% of ^{238}U and the products in the ^{238}U decay chain are given in Fig. 4.1. Any uranium sample, which is not freshly purified, will contain different radioisotopes of its the decay chain. The first decay product is ^{234}Th, which has a half life of 24.1 days. This is a convenient radiotracer for studies involving thorium and can be readily isolated from natural uranium samples. One of the separation procedures is based on anion exchange technique. In hydrochloric acid medium, hexavalent uranium forms anionic chloride complex, $UO_2Cl_4^{2-}$ while thorium forms only neutral $ThCl_4$ species. ^{234}Th can thus be separated from uranium by making a solution of uranium in 6M HCl, ensuring that uranium is present in the hexavalent state, and loading this solution on a column filled with an anion exchanger $(RN(CH_3)_3^+Cl^-)$. Under these conditions, uranium is held up on the anion exchange resin and thorium passes through along with the effluent.

$$2(RN(CH_3)_3^+Cl^- + UO_2ClO_4^{2-} \rightarrow [RN(CH_3)_3]_2UO_2Cl_4 + 2Cl^- \qquad (5.18)$$

Materials Required

Dowex 1 x 4 anion exchange resin, HCl, UO_3/UO_2, lanthanum nitrate, glass column (5 mm dia. x 10 cm), filtering setup, litmus paper, watch glass, NaOH, alcohol, mounting plates, scotch tape, cello tape, beakers and GM counter set up.

Procedure

1. Assemble the GM counter set up and make connections as given in the instruction manual. Note down the settings.
2. Switch on the power supply. After 10 minutes put on the `EHT'. Allow to stabilize for half an hour.
3. Dissolve 2 g of UO_3/UO_2 in 5 mL of 6 M HCI. Use this as a stock solution of uranium.
4. Take 3 to 4 g of Dowex 1 x 4 resin in a beaker and wash twice with water decanting the floating resin particles. Transfer the resin into an ion exchange column to form a bed volume of 5 to 6 mL.
5. Condition the column by passing 25 mL of 6 M HCI through the column at the rate of 10-12 drops/min.
6. Transfer 2 mL of uranium stock solution to anion exchange column and pass at the rate of 6-8 drops/min. Collect the effluent in a clean 100 mL beaker. Wash the column with 20 mL of 6M HCl and collect the washings in the same beaker.
7. Add 2 mL of lanthanum nitrate solution (1 mg of La/mL) to the beaker containing effluent and washings.
8. Neutralize carefully using 50% NaOH solution, checking with litmus paper. Thorium is co-precipitated with lanthanum as hydroxide. Note down the clock time.
9. Filter the solution using filter chimney unit.
10. Wash the precipitate twice with 2 mL of 0.01 M NaOH solution.
11. Wash the precipitate with 2 mL of alcohol.

12. Remove the filter paper with precipitate and dry the precipitate under I.R. lamp, if necessary. Mount the filter paper along with precipitate carefully on a mounting plate that has a double scotch paper. Cover the precipitate with a cellophane paper.
13. Place the mounting plate with precipitate in a groove of GM counter set up and count. Count the sample twice a day for 24 days under identical conditions. Note down clock time before each reading.

Observations

Date

GM Counter No. :		GM tube No. :	
Operating voltage :	V	Paralysis time :	250 μs
Source :	^{234}Th ((β,γ emitter)	Position :	Groove No.
Preset time :	60 s	Background/1800 s :	
Background/60 s (C_b) :		Step 8 Clock time :	

Table 5.8 - Counting data for ^{234}Th

Sl. No.	Start Time hh-mm	Elapsed Time (min)	Observed Counts Per 60 s (C_0)	Corrected Counts $C = C_0 - C_b$
1.				
2.				
3.				
4.				
5.				
6.				
7.				
8.				
9.				
10.				
11.				
12.				
13.				
14.				
15.				
16.				
17.				
18.				
19.				
20.				
21.				
22.				
23.				
24.				
25.				

Calculations

Plot the count rate (Y-axis) Vs elapsed time (X-axis) on a semilog paper to obtain $t_{1/2}$.

Comments and Suggestions

1. Obtain γ spectrum of ^{234}Th using NaI(Tl) with single channel analyzer.
2. Thorium-234 obtained in step 8 can be used as a tracer, due to its measurable γ activity, for studies involving thorium.
3. Uranium loaded column (step 6) can be used to milk ^{234}Th periodically.

Experiment 20

SEPARATION OF ^{234}Th FROM NATURAL URANIUM USING SOLVENT EXTRACTION PROCEDURE

Discussion

In experiment 19 an anion exchange procedure for the separation of ^{234}Th from natural uranium was described. The process was based on difference in the complexing behavior of the two actinides in chloride medium. The same principle forms the basis for separating ^{234}Th using solvent extraction. In 6M HCl medium, Tri-Lauryl amine (TLA), reacts with uranium to form $UO_2Cl_4^{2-}(TLAH^+)_2$ complex species which is soluble in $CC1_4$. Under these conditions thorium does not form organic soluble complexes. Thus uranium can selectively be separated from thorium by extracting it from 6 M HCI into CCl_4 as $UO_2Cl_4^{2-}(TLAH^+)_2$, leaving thorium in the aqueous phase.

$$UO_2Cl_4^{-2} + 2TLA.HCl \rightarrow [UO_2Cl_4^{2-}(TLAH)_2^{2+}] + 2Cl^- \qquad (5.19)$$

Materials Required

TLA, CCl_4, HCl, UO_3, lanthanum nitrate, NaOH, alcohol, filter chimney unit, separating funnel, litmus paper, watch glass, mounting plate, cello tape, scotch tape and GM counter set up.

Procedure

1. Assemble the GM counter set up and make connections as given in the instruction manual. Note down the settings.
2. Switch on the power supply. After 10 minutes put on the EHT. Allow to stabilize for half an hour.
3. Make various stock solutions as described in Experiment 19.
4. Take 2 mL of uranium from stock solution into a separating funnel. Add 5 mL of 6 M HCl followed by 20 mL of 10% TLA in CCl_4 (pre-equilibrated with 6M HCl is preferable).
5. Shake the funnel carefully for 5 min and allow the separation of the aqueous and organic phases. Drain the organic phase containing uranium into a beaker.
6. Add 20 mL of 10% TLA in CCl_4 and repeat the step 5 until the aqueous layer is colourless.
7. Collect the aqueous phase into a clean beaker.
8. Add 2 mL of lanthanum nitrate solution.
9. Neutralize carefully using 50 % NaOH solution and check with litmus paper. Thorium hydroxide will be precipitated along with lanthanum hydroxide. Note down the clock time. Filter the contents through a filter chimney unit. Wash the precipitate twice with 2 mL of 0.01 M NaOH solution. Wash the precipitate with 2 mL of alcohol.
10. Remove the filter paper with precipitate, dry the precipitate under I.R. lamp and carefully mount the filter paper along with precipitate on a mounting plate that has a double scotch paper. Cover the precipitate with a cellophane paper.
11. Count the mounting plate with the precipitate using a GM Counter as in experiment 19. Record the readings.

Observations

Date

GM Counter No.	:		GM tube No.	:	
Operating voltage	:	V	Paralysis time	:	250 μs
Source	:	La$(OH)_3$ ppt with ^{234}Th	Position	:	Groove No.
Preset time	:	60s	Background/1800 s	:	
Background/60 s (C_b)	:				

Table 5.9 - Counting data for ^{234}Th

Sl. No.	Start Time hh-mm	Elapsed Time (min)	Observed Counts Per 60 s (C_0)	Corrected Counts $C = C_0 - C_b$
1.				
2.				
3.				
4.				
5.				
6.				
7.				
8.				
9.				
10.				
11.				
12.				
13.				
14.				
15.				
16.				
17.				
18.				
19.				
20.				
21.				
22.				
23.				
24.				
25.				

Calculations

Plot the count rate (Y-axis) Vs elapsed time (X-axis) on a semilog paper to obtain $t_{1/2}$.

Comments and Suggestions

1. Obtain γ spectrum of ^{234}Th using NaI(Tl) detector with single channel analyzer.
2. Thorium-234 obtained in step 8 can be used as a tracer due to its measurable y activity for studies involving thorium-234.
3. Strip uranium from the organic phase from step 6. Keep it for six months so that ^{234}Th attains equilibrium with ^{238}U. This can be again used to milk thorium.

Experiment 21

PREPARATION OF ^{228}Ra COW FOR ^{228}Ac

Discussion

As seen in Fig. 4.2, the decay of ^{232}Th (the only naturally occurring isotope of thorium) results in the formation of a variety of radioisotopes. Among these are ^{228}Ra ($t_{1/2}$ = 5.75 y) and its daughter product ^{228}Ac ($t_{1/2}$ = 6.18 h). This experiment aims at separating Ra and Ac from natural thorium, and also preparing ^{228}Ra cow which can later be used to milk ^{228}Ac periodically. As ^{224}Ra is part of the decay chain, the isolated radium fraction will have ^{228}Ra as well as ^{224}Ra. However, ^{224}Ra is short lived ($t_{1/2}$ = 3.64 d) and most of it will decay after 3 weeks of separation. Thorium can be separated from its decay products by extraction with 30% tributyl phosphate (TBP) in kerosene. Actinium can be isolated from the raffinate by coprecipitation with lanthanum hydroxide. From Table 4.2, it is seen that after thorium has been separated only ^{228}Ra, ^{228}Ac, ^{224}Ra, ^{212}Bi and ^{212}Pb having half lives greater than 60 minutes would be present in the solution.

Materials Required

NaOH, TBP, kerosene, thorium nitrate, HNO_3, lanthanum nitrate, alcohol, filter chimney unit, separating funnel, litmus paper, planchettes, mounting plates, scotch tape, cello tape and GM counter set up.

Procedure

1. Assemble the GM counter set up and make connections as given in the instruction manual. Note down the settings.
2. Switch on the power supply. After 10 minutes put on the `EHT'. Allow to stabilize for half an hour.
3. Prepare a stock solution of thorium by dissolving 2 g of thorium nitrate in 5 mL of 4M HNO_3.
4. Take 2 mL of Th stock solution into a separating funnel and add 5 mL of 4 M HNO_3. To this, add 20 mL of 30% TBP in kerosene.
5. Shake the separating funnel carefully for 5 min to extract thorium into the organic phase. Allow the separation of the aqueous and organic phases. Reject organic phase.
6. Take the aqueous phase into another separating funnel and add another 20 mL of 30% TBP in kerosene. Repeat step 5.
7. Collect the aqueous phase in a beaker, transfer to a 25 mL standard flask and make up. This solution contains many of the products in the decay chain of thorium including ^{228}Ra, ^{228}Ac and ^{224}Ra.
8. Take the aqueous solution again in a beaker and add 2 mL of lanthanum nitrate solution. Neutralise carefully using 50% NaOH solution and check with litmus paper. The lanthanum hydroxide precipitate carries ^{228}Ac with it and leaves radium isotopes in solution. Record the clock time (T_1). Filter the contents through a filter chimney unit. Wash the precipitate twice with 2 mL of 0.01 M NaOH solution followed by 2 mL of alcohol.
9. Remove filter paper with precipitate, dry the precipitate under an I.R. lamp, and mount the filter paper along with precipitate (containing ^{228}Ac) carefully on a mounting plate that has a double scotch paper. Cover the precipitate with a cellophane paper.
10. Count the precipitate on the mounting plate. Record the reading and the clock time for each counting (T_2)
11. Step 9 can be repeated everyday to milk ^{228}Ac from the aqueous solution.

Observations

Date

GM Counter No.	:		GM tube No.	:	
Operating voltage	:	V	Paralysis time	:	250 µs
Source	:	^{228}Ra (β^- emitter) ^{228}Ac (β^- emitter)	Position	:	Groove No.
Preset time	:	60s	Background/1800 s:		
Background/60s (C_b)	:				

Table 5.10 – Counting data for ^{228}Ac

Sl. No.	Start Time hh-mm	Elapsed Time (T_2-T_1) (min)	Observed Counts Per 60 s (C_0)	Corrected Counts $C = C_0 - C_b$
1.				
2.				
3.				
4.				
5.				
6.				
7.				
8.				
9.				
10.				
11.				
12.				
13.				
14.				
15.				
16.				
17.				
18.				
19.				
20.				
21.				
22.				
23.				
24.				
25.				

Calculations

Plot the activity as a function of time and obtain the half-life of ^{228}Ac as described in Experiment 14.

Half life of ^{228}Ac =

Comments and Suggestions

^{228}Ra can be used as a cow for milking ^{228}Ac periodically.

Chapter 6

APPLICATIONS OF RADIOISOTOPES

Radioisotopes have been successfully used in the studies of a variety of problems in chemistry, medicine, industry and agriculture. Since radioisotope of an element follows the same chemistry as its stable isotopes, it can be used to trace the path of the element in physicochemical or biological processes. The applications are based on the high sensitivity in the detection of radioactive nuclides (10^{-15}g or less) and on their chemical identity with their inactive isotopes. In analytical applications, activation analysis is one of the standard tools in the measurement of trace impurities as well as major and minor constituents of a sample. Equally effective is the isotope dilution analysis for the estimation of a given element with the help of one of its radioactive isotopes. Radioimmunoassay is another technique, which is used for assaying trace quantities of hormones or drugs in the body fluids. A few experiments using radioisotope tracers, or radioactivity induced by neutron irradiation, are described in this Chapter.

1. TRACERS IN RADIOANALYTICAL CHEMISTRY

Experiment 22

DETERMINATION OF IODINE BY ISOTOPE DILUTION ANALYSIS

Discussion

A sample containing a radioisotope of an element has a certain specific activity i.e. disintegrations per unit time per unit mass of the element. If we take solution of this sample (tracer solution) and add an aliquot of a solution having unknown amount of the inactive isotope/isotopes of the element, the specific activity of the resultant sample will decrease. The extent of decrease depends on the relative amounts of the element in the tracer solution and the unknown sample[1]. Knowing the amount of the element in the tracer solution and the specific activity of the sample before and after the addition, the amount of the element in the unknown sample is determined. The advantage of this method is that the unknown sample need not be chemically pure. Quantitative separation is also not necessary.

In this experiment, the quantity of iodine in a sample is determined by isotope dilution method using ^{131}I tracer. Iodine is separated as AgI before and after dilution. Specific activities of AgI precipitates are compared to calculate the amount of iodine originally present in the sample.

Let the quantity of iodine in 1.0 mL of tracer solution be W_1 grams and the total radioactivity be A_T counts per minute (cpm). Then by definition[2], specific activity (S_1) of the iodine in the tracer solution is given by:

---

[1] This statement assumes that the decrease of radioactivity due to the decay of radioisotope is negligible. This would be true if the half life of the radioisotope is substantially longer as compared to the time required for the experiment.

[2] Specific activity is normally defined in terms of disintegration rate per unit weight e.g. dpm/g. In this experiment measured count rates are used, as the ratio of activities are taken in the calculations. Cpm and dpm are related ad spm = dpm . ε where ε is detection efficiency.

$$S_1 \text{ (cpm/g)} = \frac{A_T}{W_1}$$

$$\therefore\ A_T = S_1 \times W_1 \qquad (6.1)$$

When an aliquot of the unknown sample, having W grams of inactive iodine, is mixed with 1.0 mL of this tracer solution, there is no change in total activity (it remains as A_T). However, the specific activity of iodine in the solution decreases to S_2 as the weight of iodine in the solution is increased. S_2 is given by:

$$S_2 \text{(cpm/g)} = \frac{A_T}{(W_1 + W)}$$

$$A_T = S_2 (W_1 + W) \qquad (6.2)$$

From eqns. (.6.1) and (6.2)

$$S_1 \times W = S_2 (W_1 + W)$$

Hence the weight of iodine (W) in the unknown sample, is given by:

$$W = W_1 \left[\frac{S_1}{S_2} - 1 \right] \qquad (6.4)$$

Materials Required

^{131}I tracer (10000-20000 cpm/mL), KI solution with 10 mg of iodine/mL, $AgNO_3$ solution with 10 mg of Ag / mL, unknown KI solution to be analyzed, aluminium plates, double scotch tape, cellophane paper, Whatman 42 filter paper discs suitable for filter chimney, filter chimney unit, oven and GM counting set up.

Procedure

1. Assemble the GM counting set up and make connections as given in the instruction manual. Note down the settings.
2. Switch on the power supply. After 10 minutes put on the 'EHT'. Allow voltage to stabilize for half an hour. Count the background of the counter for 300 s and record the reading.
3. Prepare a standard tracer solution by taking 2 mL of KI solution (10 mg of iodine/mL) and adding 2 mL of carrier free ^{131}1 tracer solution (10,000-20,000 counts per minute per mL). Mix thoroughly.
4. Take two Whatman 42 filter paper discs and wash them with water followed by alcohol. Dry them in an oven at about 120° C for ten minutes and cool the filter papers outside for ten minutes. Mark them as 1 and 2, and weigh them.
5. Keep the aluminium plate fixed with double scotch tape ready for mounting.
6. Take 1.0 mL of the standard tracer solution, add a few drops of NH_4OH (pH 9) and precipitate AgI by adding $AgNO_3$ solution.
7. Separate AgI precipitate using a filter chimney unit. Complete transfer of precipitate is not a pre-requisite. Wash the precipitate with dilute HNO_3, followed by water and alcohol. Dry the precipitate (ppt_1), weigh and mount it on a mounting plate.
8. Measure the activity A_1 using a GM Counter. Record the weight and count rate.
9. Take 1.0 mL of the KI solution of unknown concentration, add 1.0 mL of the standard tracer solution and mix thoroughly. Precipitate AgI by adding $AgNO_3$ solution as in step 6 and repeat step 7 to obtain the second precipitate (ppt_2). As in step 7, complete transfer of precipitate is not a pre-requisite.

10. Measure activity A_2. Record the weight and count rate.

Observations

Date

Table 6.1 - Weighing data

Weight of	Related to Std. Tracer Soln. mg	Related to std. tracer soln. + Unknown solution mg
Filter paper		
Filter paper + AgI precipitate		
Ag I precipitate	$W_{ppt\ 1}$	$W_{ppt\ 2}$

GM Counter No. : GM tube No. :
Function : Time Preset time : 60 s
Paralysis time : 250 µs Operating voltage : V (From Experiment 1)
Position : Groove No. Sources : AgI ppt with ^{131}I
Background counts/300 s : Background count/60s (C_b):

Table 6.2 - Counting data

Sl. No.	Counts rate (per 60 s) from ppt_1	Sl. No.	Counts rate (per 60 s) from ppt_2
1.		1.	
2.		2.	
3.		3.	
Mean (A_1)		Mean (A_2)	

Calculations

Molecular weight of AgI : 235
Wt. of Iodine in ppt_1 (W_1) : $(W_{ppt1}) \times 127/235$ = mg
Specific activity (S_1) : A_1/W_1 = cpm/mg
Wt. of Iodine in ppt_2 ($W_1 + W$) : $W_{ppt2} \times 127/235$ = mg
Specific activity (S_2) : $A_2/(W_1+W)$ = cpm/mg
$W = W_1 [(S_1/S_2)-1]$ = mg
As 1.0 mL of unknown sample solution was taken,
$\therefore$ Conc. of Iodine = W/1.0 = mg/mL

Conclusions

1. Write down your conclusions on this method.
2. Comment on the versatility of isotope dilution as an analytical technique.
3. Plan an experiment, using ^{212}Pb isotope that can be separated from, natural thorium. (Discussed in Experiment 16 in Chapter 5).

Experiment 23

DETERMINATION OF THE SOLUBILITY OF A SPARINGLY SOLUBLE SALT

Discussion

Radiotracers are effectively used to determine the solubility of a sparingly soluble salt by tagging either the metal ion, or an atom of the anionic radical, with a suitable radioisotope. The labeled salt can be prepared by precipitation from a solution in which a suitable radiotracer has been added. After measuring/calculating the specific activity of this salt sample, a portion of the salt is equilibrated with water, which results in dissolution of a small fraction of the salt in water. The undissolved salt is separated and activity of the tracer in the solution is measured. These data are used for calculating the solubility of the salt. In this experiment, determination of solubility of PbI_2 by tagging iodine with ^{131}I isotope is described. This experiment can also be carried out using ^{212}Pb as a tracer, which can be prepared from natural thorium as described in experiment 16, Chapter 5.

Materials Required

Carrier free ^{131}I tracer (10^6 Bq/mL), KI solution with about 10 mg of iodine/mL, lead acetate solution with about 10 mg of lead/mL, counting tubes, transfer pipettes, cellophane paper, Whatman 42 filter paper discs suitable for filter chimney, filter chimney unit, oven and NaI(Tl) detector with accessories.

Procedure

1. Assemble the NaI(Tl) detector and single channel analyzer set up. Make connections as given in the instruction manual: Note down the settings.
2. Switch on the mains supply. After 10 minutes, apply high voltage in steps of 100 volts until rated voltage is reached. Allow to stabilize for half an hour.
3. Determine the base line voltage and window suitable to count 364 keV gamma ray (the prominent γ-ray of ^{131}I) as in Experiment 4.
4. Keep an empty counting tube in the well of NaI(Tl) detector and count for 1800 s to obtain the background counts.
5. Take 1 mL of ^{131}I tracer in a 25 mL standard flask and add 20 mL of KI solution. Make up to the mark and shake well to obtain labeled KI solution.
6. Transfer 2 mL aliquots of the above solution to two counting tubes. Count each tube thrice for 300 s to obtain counts for tracer solution (C_T).
7. Transfer 10 mL of the solution from step 5 to a centrifuge tube. Add lead acetate solution slowly to precipitate PbI_2. Test for complete precipitation by adding a slight excess of lead acetate solution.
8. Transfer the precipitate to a filter chimney apparatus having a weighed Whatman 42 filter paper. Wash thrice with 15 mL of water, dry in an oven, cool and weigh (W_p).
9. Transfer a portion of the PbI_2 precipitate from the filter paper to a centrifuge tube and add 10 mL of water. Heat slowly and shake the tube vigorously.
10. Allow the solution to cool and let the precipitate settle. Centrifuge for the clean separation of supernatant. Prepare samples by transferring 2 mL aliquots of the supernatant to two counting tubes. Measure the activity. Count each tube thrice for 300 s. This will give counts for the dissolved PbI_2 salt (C_s).

Observations

Date

Weight of PbI_2 precipitate for 10.0 mL labeled KI solution (W_p) = mg

Detector : NaI(Tl) Detector No. :
Amplifier Model : High voltage : 700 V
Mode : Differential Amplifier Gain : 3.0
Base line : V Window : V
Function : Time & Operate Sources : ^{131}I containing solutions
Position : Tube in well Preset Time : 300s
Background counts/800s : Background count/300 s (C_b) :

Table 6.3 - Counting data

Volume of the solution taken for each counting = 2.0 mL

Tube No.	Tracer			Sample		
1.	Sl. No	Counts (C_T) 300 s	Corrected Counts C_T-C_b 300 s	Sl. No.	Counts(C_s) 300s	Corrected Counts C_S-C_b 300s
	1.					
	2.					
	3.					
	Mean		(C_{T1})			(C_{S1})
2.	1.					
	2.					
	3.					
	Mean		(C_{T2})			(C_{S2})

Calculations

Wt of iodine in 2 mL of tracer solutions (W_1)	= W_P x 2/10 x 253.8/461.0	=	mg
Average counts for 2 mL of KI tracer C_1	= $(C_{T1} + C_{T2})/2$	=	counts /300 s
Specific count rate S_1	= $60C_1/300W_1$	=	cpm/mg
Average counts for 2 mL of supernatant C_2	= $(C_{S1} + C_{S2})/2$	=	counts/300 s
Average count rate for supernatant samples		=	cpm
Amount of iodine in 2 mL of supernatant	= $W_2 = C_2$ x (W_1/C_1)	=	mg
Amount of PbI_2 in 2 mL of supernatant	$(W_{LI})= W_2$ x 461.0/253.8	=	mg
Solubility of PbI_2	= W_{LI} x 50	=	g/100mL

Comments

1. Compare-this method with electroanalytical methods.
2. Repeat this experiment using a GM counter.
3. Plan an experiment using ^{212}Pb tracer instead of ^{131}I

2. NEUTRON ACTIVATION ANALYSIS

Neutron activation analysis (NAA) is a versatile and sensitive nuclear analytical technique. When a stable isotope of an element is irradiated with neutrons, some of the atoms absorb neutrons and are converted to the next isotope of mass number A+1. This reaction is represented as follows:

$$^{A}X\ (n,\gamma)\ ^{A+1}X \qquad (6.5)$$

where A is the mass number of stable isotope. Quite often the isotope ^{A+1}X is radioactive and can be used for quantitative determination of the element by measuring its radioactivity. For example,

$$^{55}Mn + n \xrightarrow[\sigma = 13.3b]{} {}^{56}Mn^{*} \xrightarrow[2.58\ h]{\beta^-} {}^{56}Fe \qquad (6.6)$$

Activity (A) formed is given by equation (6.7) and can be measured using high-resolution gamma ray spectrometry.

$$A = N\ \sigma\ \varphi\ [1 - e^{-\lambda t}] \qquad (6.7)$$

where N is number of the atoms of ^{A}X, σ is the neutron absorption cross section of ^{A}X, φ is the neutron flux, t is the time duration of irradiation and $t_{1/2}$ is the half life of radioisotope ^{A+1}X that is formed. Radioactivity of the irradiated sample can be measured by using a beta counter or a gamma spectrometer. Gamma measurement using high resolution γ-ray spectrometry is commonly used in NAA as it facilitates simultaneous multi element determination. A typical gamma spectrum of ^{56}Mn is given in Fig. 6.1.

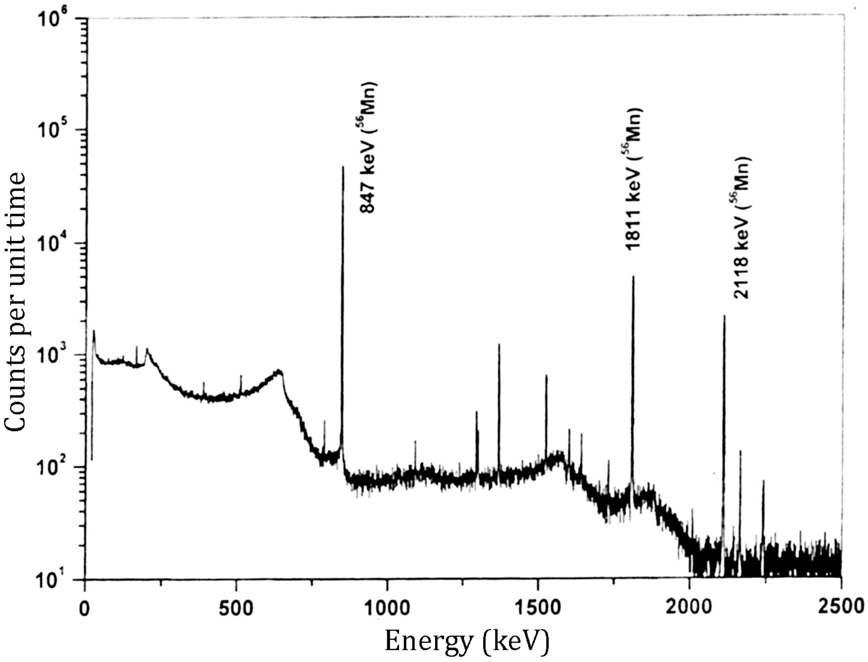


Fig 6.1 Gamma ray spectrum of ^{56}Mn.

Experiment 24

DETERMINATION OF MANGANESE IN STEEL BY NEUTRON ACTIVATION ANALYSIS

Discussion

Concentration of an element in an unknown sample can be calculated from the measured activity using eqn. (6.7). However, neutron spectra of most of the neutron sources are complex and it is not possible to assign a unique value for φ and σ. This difficulty is overcome by using a relative method.

In the relative method, a standard having a known amount (W_{std}) of the element to be analyzed is prepared. It is irradiated along with the sample under identical conditions. By replacing activity with peak area or count rate (c), eqn. (6.7) is rewritten for both standard and sample as

$$C_{sam} = N_{sam}\sigma\ \varphi\left[1 - e^{-\lambda t}\right].\ \varepsilon.\ a \tag{6.8}$$

$$C_{std} = N_{std}\sigma\ \varphi\left[1 - e^{-\lambda t}\right].\ \varepsilon.\ a \tag{6.9}$$

where C_{sam} and C_{std} are count rates of sample and standard respectively, corrected for the cooling period, ε is the detection efficiency of the γ-ray energy used and `a' is the γ-ray abundance.

Ratio of the eqns (6.8) and (6.9) gives

$$\frac{C_{sam}}{C_{std}} = \frac{N_{sam}}{N_{std}} = \frac{W_{sam}}{W_{std}} \tag{6.10}$$

Thus, weight of the element of interest W_{sam} in the sample is determined.

Materials Required

Manganese salt, thin aluminium foils, stainless steel filings, mounting plates, gamma counting set up NaI(Tl) or HPGe with analyzer) and semi-log graph papers.

Procedure

1. Assemble the NaI(Tl) detector counting set up and make connections as given in the instruction manual. Note down the settings.
2. Switch on the power supply. After 10 minutes, apply high voltage in steps of 100 V until the rated voltage is reached. Allow the voltage to stabilize for half an hour.
3. Take a known amount of manganese salt[3] in an aluminium wrapper of known weight. Wrap it properly and label it as ‘std’.
4. Prepare the sample by taking a known amount of the S.S. filings. Wrap this in another aluminium foil and label it as `sam'.
5. Keep standard and sample together in another aluminium foil and place them in an irradiation can (Fig. 6.2).
6. Irradiate the can in a position of a nuclear reactor[4] having a suitable neutron flux for a time
   duration t, to form adequate activity for obtaining good counting statistics.

---

[3] Depending on the neutron flux, weight of the manganese salt has to be decided. Typically for reactor irradiations, weight is in the range of sub mg. Hence standard is prepared by evaporating a solution of known concentration.

7. Cool the sample and the standard for 20 minutes or more depending on the activity level.
8. Calibrate γ-spectrometer using a single channel analyzer as described in experiment 6, Chapter 3.
9. Determine the base line voltage and window width suitable to count 847 keV region which is the most abundant γ ray of ^{56}Mn (see Experiment 4, Chapter 3 for details).
10. Count the standard and the sample for manganese activity[5] in a single channel analyzer using settings for 847 keV gamma ray.
11. Count the sample for ^{56}Mn activity as in step 10.

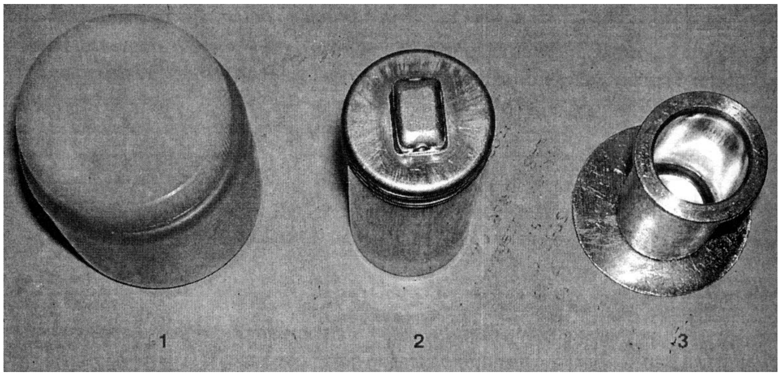


Fig 6.2 Photograph of the Irradiation Cans : (1) Polythene bottle, (2) Harwell can and (3) Cirus can (2 and 3 are made of high purity aluminium).

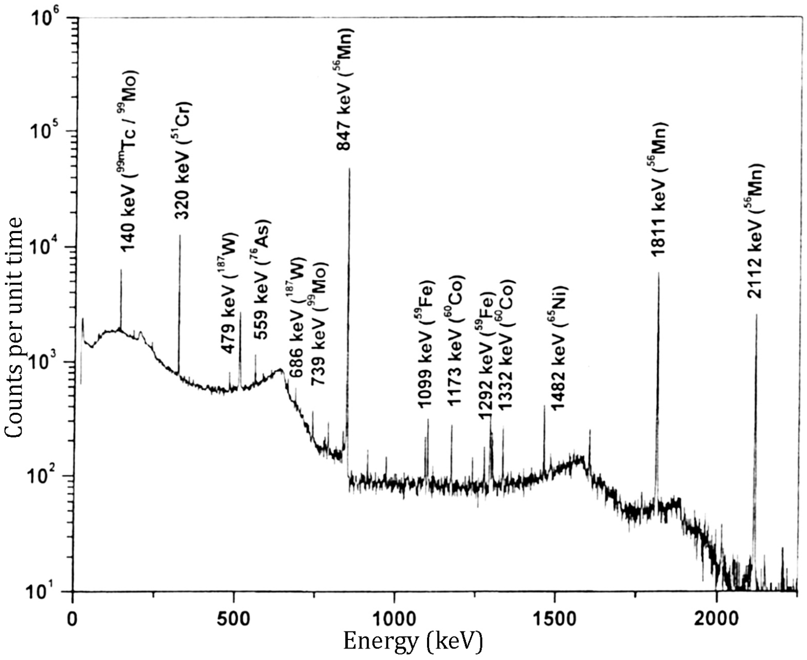


Fig. 6.3 Gamma ray spectrum of neutron irradiated stainless steel sample.

---

[4] Other neutron sources such as ^{252}Cf, Pu-Be and ^{241}Am-Be, can also be used.

[5] Gamma-spectrum of irradiated stainless steel obtained using HPGe is given in Fig. 6.3 Different γ ray peaks are marked. Intensity of these γ ray are used for simultaneous multi element analysis.

Observations .

Date

Wt. of the standard	:	% or ppm of Mn in std	:
Wt. of Mn in standard (W_{std})	:	Wt. of mild steel filing(W_{sam})	:
Wrapped and sealed in	:	Neutron source	:
Neutron flux	: n/cm^2/s		
Detector	: NaI(Tl)	Detector No.	:
Amplifier Model	:	High voltage	: 700 V
Mode	: Differential	Amplifier Gain	: 3.0
Base line	: V	Window	: V
Function	: Time & operate	Sources	: Irradiated samples
Position	: Groove No.	Preset Time	:
Background counts/1800 s	:	Background count/60 s (C_b)	:

Table 6.4 - Counting data for manganese determination

Standard				Sample			
Sl. No.	Start time	Counts / 300 s	Background Corrected countsin cps (C_{std})[6]	Sl. No.	Start time	Counts/ 300 s	Background Corrected counts in cps (C_{sam})
1.				1.			
2.				2.			
3.				3.			
4.				4.			
5.				5.			
6.				6.			

Calculations

1. Plot the activity of standard and sample separately as a function of time and check the half-lives of the activities formed.
2. From the plots, note down the activity (C^O) of both the sample and standard at zero time.

   $C^0_{sam} =$ $C^0_{std} =$
3. Calculate % of Mn in the sample using eqn. (6.10).

   $C^0_{sam}/C^0_{std} = W_{sam}/W_{std}$

   % Mn = 100 x W_{sam}/W

   where W is the weight of the sample irradiated.

Comments

1. Find out the minimum amount of manganese you can determine using this technique.
2. Comment on the versatility of this method by comparing with conventional quantitative analysis or spectrophotometric analysis.
3. What will happen if the sample contains Cr or Ni ?
4. For multielement analysis, a multielement standard has to be irradiated along with the sample. HPGe based high-resolution gamma ray spectrometry is used for measuring activities due to different radioisotopes produced. The standards can be prepared, or preferably obtained from international institutions like the International Atomic Energy Agency (IAEA), National Institute of Standards and Technology (NIST) and Institute for Reference Materials and Measurement (IRMM).

---

[6] 'C' refers to peak area and not the disintegration rate.

Experiment 25

MULTIELEMENT DETERMINATION IN SOIL BY SINGLE COMPARATOR NAA

Discussion

In a variety of materials like soil, water, cereals and biological samples, it is of interest, quite often, to determine the concentration of a wide range of elements present in the samples. Relative method of NAA described in Experiment 24 is frequently used for multi element determination. However, it requires apriori knowledge of the elements present in the samples to prepare standards suitable for analysis. Single comparator method, also known as k_0NAA method, is useful in such cases as the procedure involves the use of only one comparator (Au, Zn or Mn). This comparator is irradiated along with the sample and ratios of activities of different radioisotopes formed, with respect to the comparator are used for calculating the elemental concentrations. However, this method requires input data for many parameters. Two main inputs are the sub-cadmium to epi-cadmium flux ratio (f) and the detection efficiencies (ε) of the individual gamma rays. The concentration of i_{th} element (C_i) in the sample is calculated by using eqn. (6.11).

$$C_i(\mu g)/g) = \frac{A_p^i}{A_p^*} \cdot \frac{1}{k_0} \cdot \frac{\varepsilon^*}{\varepsilon^i} \cdot \left[\frac{f + Q_0^*}{f + Q_0^i} \right] \qquad (6.11)$$

where 'I' and '*' refer to the analyte and the comparator respectively, A_P is the peak area (specific count rate) under the characteristic γ-ray corrected for cooling and saturation, Q_0 is the ratio of infinitely dilute resonance integral cross section (I_0) and thermal neutron cross section (σ), and k_0 is a composite nuclear constant (values available in the literature) and is defined by eqn. (6.12).

$$k_0 = \left[\frac{M^* \theta^i \sigma_{th}^i \gamma^i}{M^i \theta^i \sigma_{th}^* \gamma^*} \right] \qquad (6.12)$$

where M, θ and γ are the average atomic weight of the element, the isotopic abundance and γ ray abundance respectively. Some relevant k_0 values are given in Table 6.4.

The objective of this experiment is the multi element determination in a soil sample. Accurately weighed samples of soil and gold (single comparator) are co-irradiated for 4 hours in a reactor position for which the value of 'f' is predetermined. Sample and standard are cooled for about 2 hours and assayed for γ-activity using HPGe based high resolution γ-ray spectrometer. A fixed sample to detector geometry is used for which detection efficiency as a function of γ-energy has been predetermined (Fig. 3.10, Experiment 6, Chapter 3). Peak areas under different γ-rays are compared with the peak area of 411 keV peak of ^{198}Au. From these ratios, the concentrations of different elements are obtained using eqn. (6.11)[7].

---

[7] For each set of experiments, concentrations of elements in a certified reference material (CRM) of similar matrix are determined to validate the method. For soil samples, IAEA CRM IAEA-Soil-7 is used for the validation.

Materials Required

Soil sample, standard ^{152}Eu source, thin aluminium foil, polythene sheet, gold solution standard, mounting plates and HPGe based gamma spectrometer with MCA.

Procedure

1. Assemble HPGe and the electronic units as per instructions. Note down the settings.
2. Ensure that the Dewar of HPGe detector is filled with liquid nitrogen about 6 hours before the experiment.
3. Switch on the main supply. After 20 minutes start applying high voltage at the rate of 100 V/min. After reaching the rated voltage, allow it to stabilize for half an hour.
4. Acquire γ- spectrum of a standard like ^{152}Eu.
5. Analyze for peak positions and calibrate the analyzer for energy. Note down the calibration constants and save the spectrum.
6. Open the irradiation can with irradiated sample, check for loose contamination (remove it if detected) and mount the sample on a Perspex plate.
7. Follow the same procedure for making gold comparator sample for counting.
8. Acquire the γ-spectrum of standard after placing the standard in an efficiency-precalibrated position. Label the spectrum and save.
9. Similarly acquire γ-spectrum of the sample and save the spectrum.
10. Depending upon the half-lives of the radioisotopes produced and the level of radioactivity, follow the spectrum for a few hours to a few days. Two typical γ spectra of an irradiated soil sample, at short and long cooling periods, are given in Figs. 6.4 and 6.5, respectively.

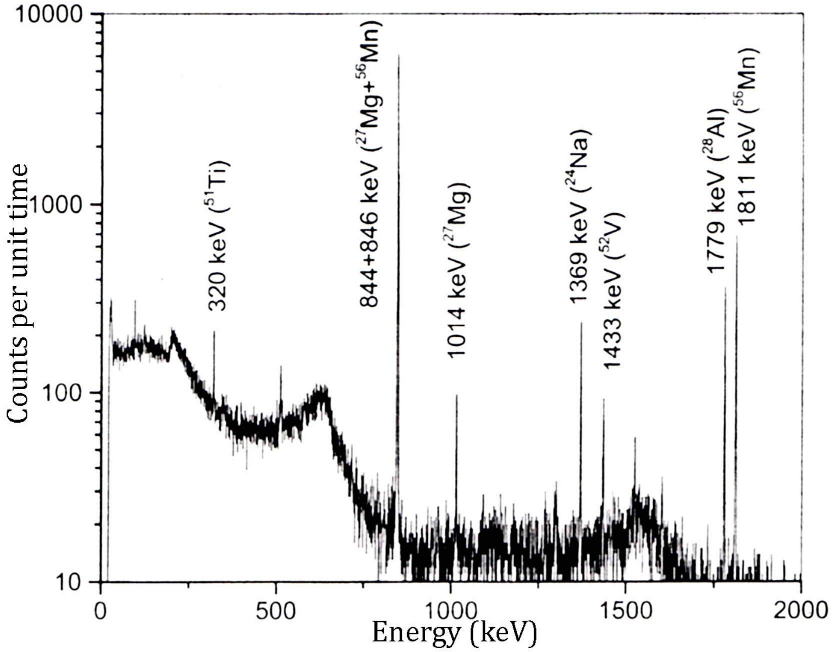


Fig. 6.4 Gamma spectrum of 5 min irradiated soil sample after 5 min cooling.

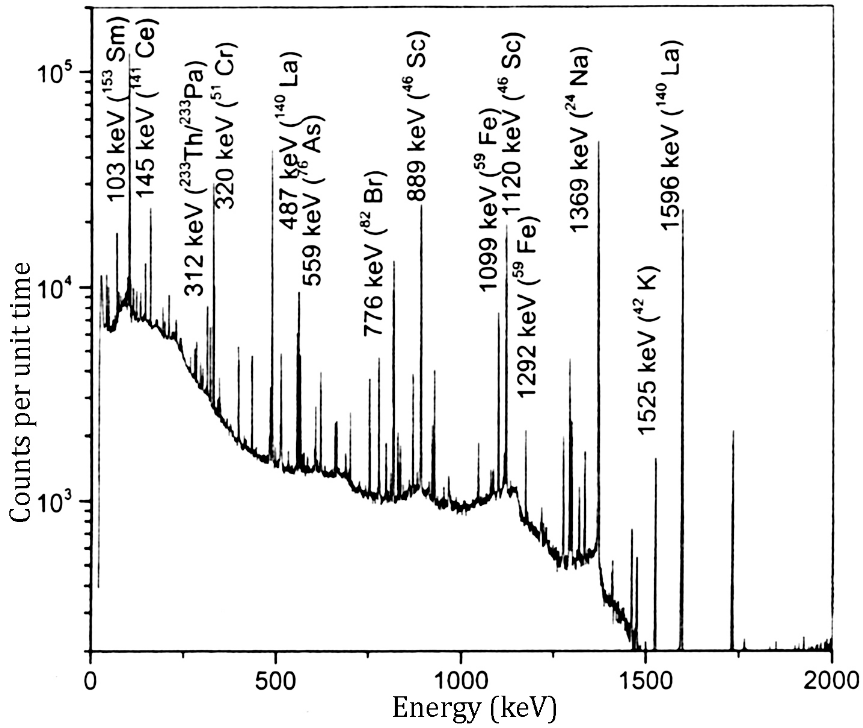


Fig. 6.5 Gamma spectrum of of 1 day irradiated soil sample after 3 days cooling.

Observations

Date

Detector	: HPGe	Amplifier Model	:
High voltage	:	Coarse Gain	:
MCA	: PHA Mode	Fine Gain	:
Calibration Standard	: ^{152}Eu	Shaping Time	: μs
Shelf number	:	Source	: Irradiated Sample

Table 6.5 - Relevant nuclear data for k_0 NAA

Element	Target Isotope	Isotopic Abundance (%)	Cross-section (σ_0 barn)	Formed isotope	Half-life ($T_{1/2}$)	Energy of the γ ray used Eγ (keV)	γ-ray Abundance (%)	$K_{0,Au}$ factors
Na	^{23}Na	100	0.512	^{24}Na	14.959 h	1368.5	100	4.68E-2
Mg	^{26}Mg	11.01	0.039	^{27}Mg	9.46 m	843.8	71.8	2.53E-4
Al	^{27}Al	100	0.226	^{28}Al	2.241 m	1779	100	1.75E-2
Cl	^{37}Cl	24.23	0.437	^{38}Cl	37.24 m	1642.4	31.9	1.97E-3
K	^{41}K	6.7	1.39	^{42}K	12.36 h	1524.7	18.08	9.46E-4
Ca	^{48}Ca	0.19	1.13	^{49}Ca	8.718 m	3084	92.1	1.01E-4
Sc	^{45}Sc	100	26.3	^{46}5c	83.8 d	889	99.98	1.22
Ti	^{50}Ti	5.18	0.174	^{51}Ti	5.76 m	320	93.1	3.74E-4
V	^{51}V	99.75	4.79	^{52}V	3.75 m	1434	100	1.96E-1

Cr	^{50}Cr	4.35	14.9	^{51}Cr	27.7 d	320.1	10.1	2.62E-3
Mn	^{55}Mn	100	13.2	^{56}Mn	2.578 h	847	98.87	4.96E-1
Fe	^{58}Fe	0.282	1.28	^{59}Fe	44.5 d	1099	56.5	7.77E-5
Co	^{59}Co	100	37.1	^{60}Co	5.27 y	1332	99.98	1.32
As	^{75}As	100	3.86	^{76}As	26.32 h	559	45	4.8E-2
Br	^{81}Br	49.31	2.58	^{82}Br	35.3 h	776.5	83.5	7.76E-2
	^{127}I	100	4.06	^{128}I	25 m	443	16.9	1.12E-2
La	^{139}La	99.91	9.39	^{140}La	40.27 h	1596	95.4	1.34E-1
Au	^{197}Au	100	98.7	^{198}Au	2.69517 d	411.8	95.56	1
U	^{238}U	99.27	2.76	^{239}U (^{239}Np)	2.356 d	277.6	14.38	3.4E-3
Th	^{232}Th	100	7.34	^{233}Th (^{233}Pa)	26.97 d	312	38.6	2.52E-2

Table 6.6 - Counting data

Sl. No.	Start time hh-mm	Counting time (min)	Peak areas corresponding to different gamma rays (keV)					
			1368.5	843.8	1779	1642.4	1524.7	3084
1								
2								
3								
4								
5								
6								
7								
8								
9								
10								
11								
12								
13								
14								
15								
16								

Table 6.6 - Counting data (contd)

Sl. No.	Start time hh-mm	Counting time (min)	Peak areas corresponding to different gamma rays (keV)					
			884	320	1434	847	1332	559
1								
2								
3								
4								
5								
6								
7								
8								
9								
10								
11								
12								
13								
14								
15								
16								

Calculations

1. Analyze all γ-ray spectra for peak areas. Correct the peak areas for decay, cooling and saturation to obtain normalized count rates (A_p^i) for all the energies of γ-rays used.
2. Similarly calculate the normalized peak area for the standard (A_p^*). Calculate concentrations of all elements in the sample using eqn. (6.11) and enter them in the last row (16) of the Table 6.6.
3. Detection efficiency values are to be taken from predetermined calibration plot.

3. AGRICULTURAL RESEARCH

Experiment 26

IODIDE UPTAKE BY PLANTS

Discussion

Iodine is one of the prominent fission products. Total fission yield of all iodine isotopes is 8-12% in the fission of uranium and plutonium, which are used as fuel for nuclear power production. It is normally contained in the nuclear fuel and its cladding. During abnormal incidences involving a steep rise in the temperature of the nuclear fuel, coupled to a breach in clad material, small quantities of iodine may be released to the environment by volatilization and escape through the ventilation system of the reactor. A constant vigil is kept on the release of radioisotopes through the ventilation system. Fission produced iodine consists of many isotopes from ^{128}I to ^{141}I, of which ^{131}I ($t_{1/2}$ = 8.04 d) is most prominent. Iodine is an important micronutrient and, therefore, radioiodine can readily enter the life cycle through food chain if released into environment. Iodine is very important in the functioning of the thyroid gland of the human system and most of the iodine in our food reaches this gland. Radioactive iodine, if present in food, would accumulate in this gland.

The present experiment aims at studying the iodine uptake in the form of I^- by root, shoot and plant as a whole in its initial stages of growth from soil and the depletion of iodine from soil. Wheat has been chosen for this study because of its wide distribution and consumption. Since it belongs to grass family, information obtained can be correlated to the iodine entry into human body through the food chain vis-a-vis grazing by animals and to humans through animal products.

Materials Required

Wheat seeds (200 grains), washed sand in a beaker (1 liter capacity), standard nutrient solution (Steinberg solution), ^{131}I activity (10 µCi), standard KI solution (50 mg/mL), well type NaI (Tl) detector with single channel analyzer set up, ^{241}Am and ^{137}Cs sources, counting tubes (20 nos.) and parafilm.

Procedure

1. Fill the 1 liter beaker with washed sand up to 3 quarters of its height. Add nutrient solution.
2. Take 10 µCi of ^{131}I activity in a 100 mL flask and make up the volume with standard KI solution.
3. Take 50 µL solution from the above flask in a standard counting tube, make-up to 2 mL with water and seal the tube with parafilm. This tube containing 50 µg of KI will be the standard.
4. Mix the rest of the labeled KI solution with the sand in the beaker. Add water to wet the sand and mix thoroughly. Take this soil (sand) and fill in a weighed counting tube up to 2 mL mark. Weigh the tube after drying the sand in an oven. This sample is the soil standard.
5. Spread washed wheat seeds (about 100) evenly just below the soil and keep the beaker preferably in sunlight for a few hours every day. Water the seeds daily to keep the surface of the sand just moist.

6. After about 5 to 8 days, the shoot will start appearing. From this day onwards pluck out carefully about 5 plants every alternate day. Wash the roots completely with water. Dry the plants with absorbent sheets.
7. Cut and separate the shoots from roots and, weigh both shoots and roots. Fill the shoots and roots in two separate counting tubes. See that the sample height in the counting tube is below 2.5 cm.
8. Count the activity of ^{131}I in shoots, roots and the standard labeled KI solution (from step 3).
9. Take out the soil (sand) from the top layer 16 to 20 days after shoots first appeared, fill in a weighed counting tube, dry in an oven as in step 4 and weigh again.
10. Count the ^{131}I activity in the soil before (step 4) and after the experiment (step 8).
11. As in step 9 take the weighed amount of soil from the bottom of the beaker in a counting tube and count the activity.

Observations

Date of sowing the seeds :
Date of first observation of shoots (Call this as zeroeth day) :
Strength of standard labeled KI solution : ~50 µg of I^-/mL

Detector	:	NaI(Tl) well type	Detector No.	:	
Amplifier Model	:		High voltage	:	700 V
Mode	:	Differential	Amplifier Gain	:	
Base line	:	V	Window	:	V
Function	:	Time & Operate	Sources	:	^{131}I
Position	:	Tube in well	Preset Time	:	100 s
Background:			Background :		
counts/1800			count/60 s (C_b)	:	

Table 6.7 - Counting data of solution standard and shoots/roots

Day	Standard			Shoots			Roots		
	Wt. µg I	C/100 s	C/ µg I	Wt. mg	C/100 s	µg I/mg	Wt. mg	C/100 s	vig I/mg
0									
2									
4									
6									
8									
10									
12									
14									
16									
18									
20									

Table 6.8 - Counting data of soils

Sample Type*	Wt. of Sample g	Counts 100s	Counts/g 100s
Soil standard			(C_{soil})
Depleted top soil			(C_{DPS})
Depleted bottom soil			(C_{DBS})

* All samples should be counted within 15-20 minutes.

Calculations

1. From the count rate of the standard labeled solution, calculate the count rate per 100 s per µg of I^- as a function of time (see table).
2. From the count rates, determine the quantity of iodine in shoots/roots.
3. Estimate the iodine uptake per mg of roots and shoots and total plant (add total uptake by shoots and roots and then divide by the total weight).

$$\text{Uptake} = \frac{\text{Wt of } I^-(\mu g)}{\text{Wt of plant or plant part (mg)}} \qquad (6.13)$$

4. Calculate the transport index as a function of growth of the plants.

$$\text{Transport Index} = \frac{100 \text{ x Wt. of } I^- \text{ in shoots } (\mu g)}{\text{Wt. of } I^- \text{ in shoots } (\mu g) + \text{Wt. of } I^- \text{ in roots } (\mu g)} \qquad (6.14)$$

Table 6.9 - Data on iodine uptake by plant and plant parts

Day	Uptake by shoots	Uptake by roots	Uptake by plants	Transport Index
1				
2				
4				
6				
8				
10				
12				
14				
16				
18		.		
20		.		

4. Plot the activity in shoots, roots and total plant as a function of growth. The plot is expected to similar to Fig. 6.6.
5. Calculate the percent depletion of iodine in the soil used for this experiment from the initial and final activity per gram of top soil. Assume that the initial ^{131}I activity in top and bottom levels to be same. From the final activity in bottom soil, calculate the % depletion (or migration) at bottom.
   Percent depletion of top soil =
   Percent depletion of bottom soil =

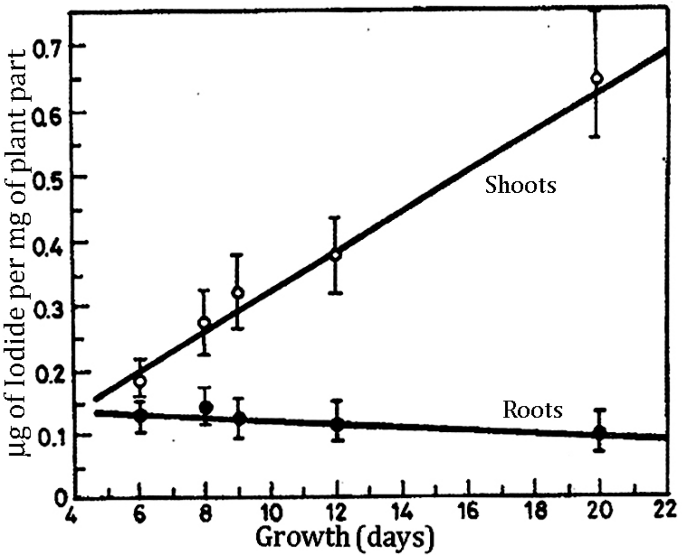


Fig. 6.6 Uptake of iodine in T in shoot and root as a function of growth.

3. RADIOPHARMACY

Experiment 27

DETERMINATION OF THYROXINE (T4) BY RADIOIMMUNOASSAY

Discussion

Radioimmunoassay (RIA) is a widely used analytical technique to measure the levels of biomolecules such as hormones present at subpicomole quantities in complex matrices such as human serum, urine and tissue extracts. The analyte is often called an antigen (Ag). This technique is based on the reaction between the antigen (Ag) and its specific antibody (Ab), which can be followed by using a radiolabeled antigen (Ag*) as a tracer. RIA is a highly sensitive and specific technique. The sensitivity is achieved due to the use of radiotracers which can be detected at very low levels and the specificity is imparted by the high affinity constant of the Ag-Ab reaction. Antibodies are molecules (e.g. gamma gloubulins, M.Wt. 150,000), produced by the immune system of animals or humans in response to the presence of a foreign moiety (the antigen), in its body. The antibodies are very specific and when in contact with the specific antigen they form a complex with a great affinity. RIA technique for the determination of an Ag requires a radiolabelled antigen and a specific antibody. If a mixture of unlabelled and radiolabelled antigen are allowed to react with a limited amount of the antibody, then both the radiolabelled and unlabelled analyte compete for the limited binding sites on the antibody molecules. Hence, if the amount of antibody and the radiolabelled antigen are kept constant, the probability of the radiolabelled antigen to form the complex will decrease with increasing amount of unlabelled antigen. A calibration curve is obtained by plotting radioactivity in the bound fraction as a function of the concentration of the antigen. The concentration of analyte in the sample is obtained from this curve. Determination of thyroid hormones like T_3, T_4, and TSH is carried out routinely using RIA and determination of T_4 is described in this experiment. When a mixture of normal T_4 hormone and radiolabelled T_4 hormone are reacted with a limited quantity of a T_4 specific antibody, then the unlabelled T_4 competes with radiolabelled T_4 for the limited binding sites on the antibody made specifically for T_4. At the end of the reaction, the T_4 bound to antibody (Ag-Ab) and free T_4 are separated by the addition of polyethylene glycol (PEG), which precipitates the Ag-Ab complex. Counting of the precipitate yields a value for the percentage of labelled T_4 bound to the antibody. This is plotted against the concentration values of the T_4 standards to obtain a calibration curve as shown in Fig 6.7. T_4 concentration in the patient sample can be calculated by interpolation of the percent bound value for the sample. This test can be performed using as low as 10 µL of the serum and the assay covers the range of 0-200 ng/mL of T_4.

Materials Required

Table 6.10 - Reagents provided in the RIA Kit

Sl. No.	Reagent	RIAK-5A 100 ASSAY TUBS
1.	T_4 standard (lyophilized)	1 vial
2.	T_4 antiserum (lyophilized)	1 vial
3.	^{125}I labeled T_4 (lyophilized)	1 vial
4.	T_4 free human serum (lyophilized)	1 vial
5.	Control serum A and B (lyophilized)	1 vial each
6.	Concentrated assay buffer (solution)	1 vial
7.	Polyethylene glycol (dry powder)	1 packet

Other reagents: Reconstitution reagents. Reconstitution of Reagents

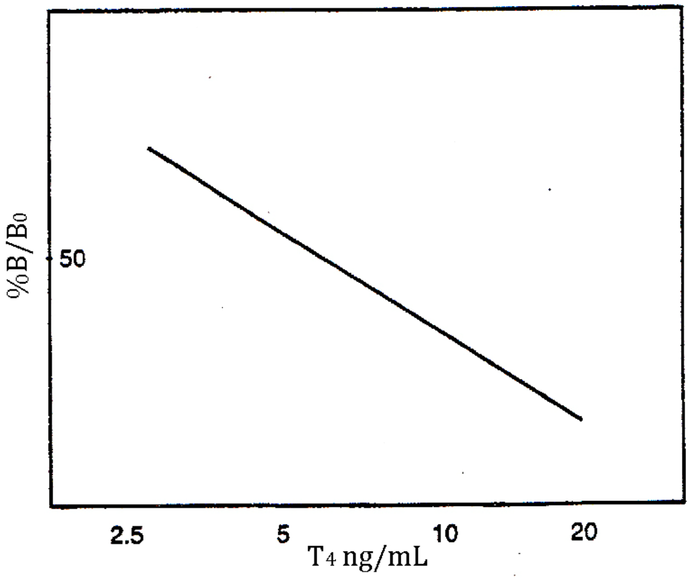


Fig. 6.7 Model curve T_4 standard curve on logit-log scale.

Reconstitution of Reagents

Decap the reagents carefully since they are sealed under vacuum. Do not interchange or discard the rubber closures. Lyophilized and dry powder reagents are to be reconstituted as described below. Store all the reconstituted reagents at 4°C. At this temperature the reagents are stable for one week.

(i) Assay Buffer

Add 100 mL double distilled water to the contents of the concentrated buffer vial. This reconstituted assay buffer contains 0.1 % gelatin in 0.14 M THAM (Tris-Hydroxymethyl Amino Methane) buffer, pH 8.6.

Note: Concentrated buffer vial, if stored at 4°C, should be kept at 37°C for 5 minutes before dilution.

(ii) T_4 standards

Add 4 mL of assay buffer to the standard. Mix gently. T_4 hormone concentration in the reconstituted standard is 20 ng/mL and is called standard A. Prepare other standards from standard A as given below.

Sl. No.	Standard	B	C	D
1.	Assay buffer in mL	0.5	1.5	3.5
2.	Standard A in mL	0.5	0.5	0.5
3.	T_4 concentration (ng/mL)	10.0	5.0	2.5

(iii) T4 antiserum (serum containing antibodies specific to T_4): Add 10 mL of assay buffer and mix gently.
(iv) $^{125}I\text{-}T_4$: Add 10 mL of assay buffer and mix gently.
(v) T_4 free serum: Add 5 mL of assay buffer and mix gently.
(vi) Control sera A and B (C_A and C_B): Add 5 mL of assay buffer to each vial and mix gently.

(vii) Transfer PEG powder to a 125 mL reagent bottle containing 90 mL of 1 % (w/v) NaCl. Mix it well till dissolution is complete.

Observations

Date

Detector	:	NaI(Tl) well type	Detector No.	:	
Amplifier Model	:		High voltage	:	700 V
Mode	:	Differential	Amplifier Gain	:	
Base line	:	V	Window	:	V
Function	:	Time & Operate	Sources	:	^{125}I
Position	:	Tube in well	Preset Time	:	100 s
Background:			Background :		
counts/1800			count/60 s (C_b)	:	

Procedure

1. Assemble the electronic units of the RIA counter NaI(Tl) based single channel analyzer and make connections as per instructions. Fix bias voltage and window for counting gamma rays from ^{125}1. Note down the settings. Switch on the mains supply. After 10 minutes put on the 'EHT'. Allow to stabilize for half an hour.
2. Take 50 µL of each control serum and sample with unknown concentration in tubes labelled serially as S_1, S_2, etc. Dilute each solution to 500 µL with assay buffer.
3. Take eighteen assay tubes and label two of them as T_1, T_2 and the rest with numbers 1 to 16. Add 2 tubes more per sample for unknown samples. To each of these tubes add the reagents using a micropipette as given in Table 6.11.

Table 6.11 - Volumes (µL) of reagents to be added in different tubes

Tube No.	Buffer	Fee serum	Std sample (1:10)	^{125}1-T_4	Antiserum
T_1, T_2 (Total)	-	-	-	100	-
1,2 (NSB*/Blank)	200	100	-	100	-
3,4 (Zero)	100	100	-	100	100
5,6	-	100	100 (D)	100	100
7,8	-	100	100 (C)	100	100
9,10	-	100	100 (B)	100	100
11,12	-	100	100 (A)	100	100
13,14	100	-	100 (S_1/C_A)	100	100
15,16	100	-	100 (S_2/C_B)	100	100

*NSB = Non-specific binding

4. Mix the tubes gently and incubate either at 37°C for 30 min or at room temperature for 75 min.
5. Keep tubes T_1 and T_2 aside and add 1.0 mL of PEG solution to all other tubes (1 to 16).
6. Mix and centrifuge at about 2000 G for 20 min. ($G = (RPM)^2 \times r \times 1.18 \times 10^{-5}$ where r = radius of the rotor in cm).
7. Discard the supernatant. Carefully remove the last traces of the supernatant with a wick cut out from an absorbent sheet, without touching or disturbing the precipitate.

8. Count all the tubes using a Nal(TI) single channel analyzer with window set for ^{125}1.

Observations and Calculations

1. Obtain average counts per minute (cpm) for all duplicate tubes.
2. Subtract counter background from the average counts of total (T_1, T_2) to get corrected total counts and from the average counts of blank/NSB (Tube Nos.1, 2) to get corrected blank/NSB counts.
3. Calculate %Blankt/%NSB

$$\%\text{Blank}/\%\text{NSB} = \frac{\text{Corrected average counts of tube No.1 \& 2}}{\text{Corrected average total counts}} \text{ x } 100 \qquad (6.15)$$

%Blank/%NSB should be less than 10% of total. Subtract these counts from the average counts of all the remaining duplicate counts.

4. Calculate zero binding (%B/B_0)

$$\%B_0/T = \frac{\text{Corrected average counts of tube No. 3 \& 4}}{\text{Corrected average total counts}} \text{x}100 \qquad (6.16)$$

This value should be less than 100 to ensure that the amount of antibody available is less than the amount of antigen taken. Generally antibodies that will result in ~50% binding are used. However, in the case of T_4 assay, the value should be greater than 80% to ensure desired wide range.

5. Calculate %B/Bo

$$\%B/B_0 = \frac{\text{Corrected average counts of std. or sample}}{\text{Corrected average counts of tube No. 3 \& 4}} \text{ x } 100 \qquad (6.17)$$

Typical counting data along with the above calculations are given in Table 6.12.

6. Plot the corrected average counts against the concentration of T_4 (/mL) on a semilog paper to obtain a sigmoid curve or plot %B/B_0, on a logit scale against concentration of T_4 (ng/mL) on a log scale of logit-log graph paper to obtain a straight line.
7. Read the sample value from the calibration curve. T_4 concentration of the sample can be expressed as:
   a. ng/mL by multiplying the value by 10 (because of initial dilution).
   b. µg/dL by simply reading the value from the standard curve.

   A typical model calculation is given in Table 6.12

Table 6.12 - Typical model calculation for T_4 assay. (Counter background = 433 cpm)

Content	Tube No.	Count rate, cpm	Corrected average counts	%B/B0
Background		50	-	-
Total	T_1, T_2	63591, 64432	63962 (T)	-
Blank / NSB	1,2	1494, 1397	1396	-
Zero Std	3,4	34024, 34534	32833 (B_0)	100
Standard D	5,6 (2.5 ng/mL)	23351, 23962	22211	67.7
Standard C	7,8 (5 ng/mL)	17158, 17205	15736	47.9
Standard B	9,10 (10 ng/mL)	12116, 11930	10577	32.2
Standard A	11,12 (20 ng/mL)	7542, 7354	6002	18.3
Sample 1 S_1 (Control A)	13,14	16525, 15557	14595	44.5
Sample 2 S_2 (Control B)	15,16	10570, 10685	9182	28.0

NOTE: % Blank / % NSB = 2.2%; % Zero binding = 67.7%

The sample values (S_1, S_2) read from the std. curve.are 60 ng/mL (S_1) and 120 ng/mL (S_2)

Table 6.13 - Counting data for assay of T_4 hormone

Background = cpm

Content	Tube No.	Count rate, cpm	Corrected average counts	%B/B_0
Background	-			
Total	T1, T2			
Blank / NSB	1,2			
Zero Std	3,4			
Standard D	5,6 (2.5 ng/mL)			
Standard C	7,8 (5 ng/mL)			
Standard B	9,10 (10 ng/mL)			
Standard A	11,12 (20 ng/mL)			
Sample 1 S_1 (Control A)	13,14			
Sample 2 S_2 (Control B)	15,16			

Experiment 28

OPERATION OF A ^{99}Mo - ^{99m}Tc GENERATOR AND PREPARATION OF RADIOPHARMACEUTICALS

Discussion

^{99m}Tc is the radioisotope of choice for in-vivo nuclear medicine procedures. It has a short half-life of six hours and emits gamma rays of moderate energy (140 keV, 90% abundance). ^{99m}Tc is the daughter product of ^{99}Mo and the decay chain is as follows:

$$^{99}\mathrm{Mo} \xrightarrow[66\ \mathrm{h}]{\beta^-} {}^{99m}\mathrm{Tc} \xrightarrow[6\ \mathrm{h}]{\mathrm{IT}} {}^{99}\mathrm{Tc} \qquad (6.17)$$

This system is an example of transient equilibrium and ^{99m}Tc can be milked, periodically from a ^{99}Mo - ^{99m}Tc generator.

Column chromatography over acidic alumina and solvent extraction using methyl-ethyl ketone are the most commonly used methods for radiochemical separation of ^{99m}Tc from ^{99}Mo. The former procedure is the most popular due to its simplicity and ease of operation at the users end. However, for large-scale preparation of ^{99m}Tc, for use in nuclear medicine centres in our country, BARC's solvent extraction generator is used. In this experiment, a ^{99}Mo generator system will be operated and the purity of ^{99m}Tc prepared will be evaluated. A common ^{99m}Tc radiopharmaceutical preparation will be made using a kit supplied by Board of Radiation and Isotope Technology (BRIT) and its quality evaluated.

Materials Required

^{99}Mo as sodium molybdate, 0.9% (w/v) NaCI (saline), acidic alumina, glass column with sintered glass filter (G-2), nitric acid, Whatman chromatography paper and a kit[8] for ^{99m}Tc-DTPA complex preparation.

Procedure

1. Assemble the electronic units of a NaI(Tl) counting setup and make connections as given in the instruction manual. Note down the settings.
2. Switch on the mains supply. After 10 minutes put on the 'EHT'. Allow to stabilize for half an hour.
3. Take about 5 g of acidic alumina and suspend in water. Remove the fine particles and condition the alumina with an acid solution at pH 3-4. Transfer the material to a glass column having a sintered glass filter (G-2).
4. Take the ^{99}Mo solution (normally supplied as alkaline sodium molybdate). Add HCI to adjust pH 3 and load on the alumina column.
5. Wash alumina column with about 100 mL of normal saline (0.9% w/v). Collect the washings in evacuated vials and treat as radioactive waste. The column is now ready for use as a ^{99m}Tc generator.
6. Elute ^{99m}Tc with 10 mL of normal saline solution using an evacuated vial. ^{99m}Tc can be eluted once in a day as sodium pertechnetate.
7. Check the $^{99m}\mathrm{TcO}_4^-$ eluted for its radioactivity content and the radiochemical purity[9] as follows:

---

[8] Kits for the preparation of ^{99m}Tc radiopharmaceuticals contain pre-mixed reagents at required concentrations and pH. These reagents when mixed with the generator produced Tc in the form of TcO_4^- yield the desired radiochemical formulation. Since the organ specificity is determined by the radiochemical form, the kits are handy for the preparation of specific radiopharmaceuticals.

[9] Determine the radiochemical purity of the eluted $^{99m}\mathrm{TcO}_4^-$ by paper chromatography (solvent 85% methanol, Whatman 1, R_f of TcO_4^- 0.4 to 0.5). The radiochemical purity required is 90%. This pertechnetate solution is used for preparing the radiopharmaceuticals.

a. Prepare a source using an aliquot of the eluted ^{99m}Tc and analyze using a NaI(Tl) detector with a single channel analyzer. Compare with a standard to determine the ^{99m}Tc radioactivity in the aliquot.
b. Take a γ-spectrum to check the absence of ^{99}Mo (Eγ = 740 keV). The permissible limit for ^{99}Mo for medical use is 0.3%.
c. Determine the radiochemical purity as the percentage of the ^{99m}Tc radioactivity in the stated chemical form i.e. as pertechnetate.

8. Preparation of 99mTc-DTPA complex:

^{99m}Tc-DTPA is a radiopharmaceutical used for renography. A lyophilized kit containing DTPA (35 mg), $SnC1_2.2H20$ (2mg) and NaCl, is provided. Add 3 mL of the $^{99m}TcO_4^-$ eluted from the generator to the kit vial using a syringe. Mix well. Keep the vial in a boiling water bath for 5 min. Cool the contents and the product is ready for use.

9. Quality control:

The impurities expected in this product are reduced/hydrolysed Tc (TcO_2) and free TcO_4^-. An ascending paper chromatography is done for estimating the radiochemical purity of the ^{99m}Tc-DTPA prepared. $^{99m}TcO_4^-$ is also analyzed along with the complex on another strip to serve as a reference. Chromatographic paper Whatman 1 (2.5 x 20 cm) and normal saline are used for this purpose. Develop the chromatography strips up to the top of the paper. Remove the paper. dry and cut it into 1 cm segments. Count each segment in a NaI(Tl) scintillation counter. R_f values are as follows:

^{99m}Tc-DTPA: 0.9-1.0
Reduced/hydrolysed Tc : 0.0
$^{99m}TcO_4^-$: 0.6-0.7

The radiochemical purity of the product (Tc-DTPA complex) should be above 90%.

Observations & Calculation

1. Radioactive content of eluted $^{99m}TcO_4^-$

Sample volume = µL (a)
Activity expected in the standard = kBq (b)
Count rate of sample = cpm (x)
Count rate of standard = cpm (y)
Radioactivity in the sample = [(x/y) b] kBq = kBq
Radioactive Concentration = (x1y) (b/a) kBq µL^{-1} = MBq mL

2. ^{99}Mo content in eluted ^{99m}Tc

Peak area at 740 keV = cpm
Amount of ^{99}Mo in this aliquot = kBq
% ^{99}Mo in eluted ^{99m}Tc =

3. Radiochemical purity of eluted TcO_4^-

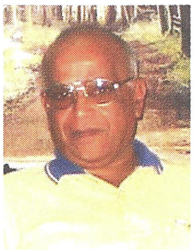
(a) Total activity loaded = cpm
(b) Activity at R_f 0.4 - 0.5 = cpm
(c) % Radiochemical purity =

4. Radiochemical purity of 99mTc - DTPA complex

(a) Total activity loaded = kBq
(b) Activity at Rf 0.9-1.0 = kBq
(c) % Radiochemical purity of the complex =

About the Authors

Dr. D.D. Sood, after successful completion of one year orientation course from Training School of the Bhabha Atomic. Research Centre (BARC) in 1959, joined Radiochemistry Division, BARC, Mumbai. He studied cation diffusion in uranium and thorium oxides at Imperial College of Science and Technology, London during 1966 and worked on the molten salt reactor concept at Oak Ridge National Laboratory, USA during 1970-72. He is an expert in chemistry and metallurgy of plutonium with special reference to plutonium based fuels. He has published more than 150 research papers and 25 technical reports. Fifteen students have obtained Ph.D. under his guidance. He is a member of the Advisory Board of the Journal of Chemical Thermodnyamics. He has held the designations of Head, Fuel Chemistry Division, BARC and Director, Radiochemistry and Isotope Group, BARC. He served as Director, Division of Physical and Chemical Sciences, IAEA, Vienna during 1998-2003. He was president IANCAS for two terms. He has authored/coauthored 3 other books. He is recipient of many awards including IANCAS Dr. M.V. Ramaniah Memorial Award for the outstanding and life time achievements in the field of Nuclear and Radiochemistry.



Dr. S.B. Manohar formerly Head, Radiochemistry Division, BARC, obtained M.Sc. in Chemistry from University of Pune, Pune and completed one year orientation course from BARC training school during 1965-66. He joined Radiochemistry Division and since then has been actively engaged in programmes of Nuclear Chemistry. His areas of research are nuclear fission, nuclear reactions, radioactivity in the environment, radiochemical separations, neutron activation analysis and development of non-destructive assay methods. He has published more than 150 scientific publications. He is a member of many scientific associations and served IANCAS as vice president (1997-2000) and president (2000-03). He served as an IAEA expert on environmental radioactivity. He is the recipient of IANCAS Dr. M.V. Ramaniah Memorial Award for the life time achievements in the field of Nuclear and Radiochemistry..

Dr. A.V.R. Reddy after graduating from BARC Training School in 1976-1977, joined Nuclear Chemistry Section, Radiochemistry Division, RC&IG and was its Head from 1998. He is at present Head, Analytical Chemistry Division, Chemistry Group, BARC. His main areas of research are nuclear fission, nuclear reactions, radiochemical separations, neutron activation analysis, electrochemistry and general analytical chemistry. He has about 600 publications in journals and symposia. He is a co-author of the books, Fundamentals of radiochemistry, Experiments in Radiochemistry, Introduction to Radiochemistry and Experiments in Analytical Chemistry. He is a Professor of Homi Bhabha National Institute. He has worked as a visiting scientist for an year on the extension of Periodic Table in Institut für Kernchemie, Mainz, Germany. Dr Reddy served as a Technical Officer during 1999-2000 in the Division of Physical and Chemical Sciences, IAEA, Vienna. He is a fellow of IUPAC (International Union of Pure and Applied Chemistry) and was a commission member of IUPAC's Commission on Radiochemistry and Nuclear Techniques during 1996-2002. He served as an IAEA expert and visited many countries to provide advice on nuclear analytical techniques and radiochemistry. Dr. Reddy was Member Secretary, Basic Science Committee, BRNS (2010-2014) and member, SERB PAC, DST. He served as Secretary (1991-93 & 2009-11), and currently he is Vice President, IANCAS and Vice President, AEACI.