CHAPTER 3 : INSTRUMENTATION

3.1. Introduction

Radiation can be classified into two groups viz particulate and electromagnetic. These electromagnetic radiations can be further grouped as non ionizing radiation such as visible light, and ionizing radiation such as γ rays and X rays. Ionizing radiation has the ability to ionize the atoms and the molecules of the media it passes through. Particulate radiation can be classified into charged radiation such as α and β particles and uncharged radiation such as neutrinos and neutrons. The backbone of studying environmental radioactivity and radioecology is radiation detection and radioactivity analysis. The radiation detectors, which include the detector, the signal-processing unit and the output display such as counter or spectrometer. The basic working principle of the various nuclear radiation detectors is similar. Radiation enters the detector, interacts with the atoms of the detector material losing part or whole of its energy and releases a large number of relatively low energy electrons from the orbits of atoms. The present study aims at the estimation of radioactive substances present in the environmental matrices. A brief description of the detectors used in the experiment is given below.

3.2. Scintillation Radiation Monitor

The survey meters based on GM detector have been used by many researchers like Paul et al to perform radiation survey at high background area in Kerala, India and Ghiassi-nejad et al in Ramsar, Iran. Also some of the investigators like Ameral et al., 1992; Eisenbud and Petrow, 1964; Fernandez-Aldecoa et al., 1992 and Sunta et al., 1982 have used the GM survey meter due to their advantages like lightweight and simple electronics [39, 192-196]. However, the main disadvantages of these detectors are their sensitivity and internal background. Because of the poor efficiency and the consequent low count rate, long time constants were required for accurate measurements. Hence, they are suitable for stationary measurements. Scintillation detectors are more sensitive compared to the survey meters featuring GM detectors. Portable high-sensitivity Radiation Monitors are designed to detect sources of gamma radiation and measure ambient gamma radiation dose equivalent rate from 30 nSvh⁻¹
to 300 $\mu$Sv h$^{-1}$. Scintillation radiation monitor is a lightweight portable radiation survey meter, featuring solid-state design and ideally suited for radiometric, geophysical and environmental surveys.

![Block diagram of Scintillation Radiation Monitor](image)

Figure 3.1: Block diagram of Scintillation Radiation Monitor

**Working:**

The detector is a thallium activated sodium iodide crystal of dimension 25×40 mm, optically coupled to a photo multiplier. The crystal and the photomultiplier assembly are hermetically sealed. The block diagram and the full view of the scintillometer are as shown in Figure 3.1 and Figure 3.2 respectively. Scintillator, the detecting element coupled with the photocathode will send electrical pulses resulting from radiation exposure. After proper discrimination, the pulses are parallely routed to dose rate counting network and cumulative dose network through a 12/13 divider and is being counted. The instrument directly gives the digital displays of the exposure rate. Thus, the instruments allow high-accuracy dose rate measurement in a wide range of gamma energies due to "Spectrum-Dose" correction functions in the energy range from 0.05 to 3 MeV. The specifications of the radiation monitor are tabulated in Appendix I.

![Photographic view of Scintillation Radiation Monitor](image)

Figure 3.2: Photographic view of Scintillation Radiation Monitor
3.3. Alpha Counting System

Historically Ionization chambers and later proportional counters were used for the detection of alpha particles and presently scintillation counters with silver activated Zinc Sulphide phosphor and doped semiconductor detectors are almost universally used. Since alpha particles have very high specific ionization, which greatly exceeds most of the cosmic ray components, good counter discrimination against natural background radiation can be achieved simply by voltage bias adjustments or by the choice of the physical dimensions of the detector to preferentially absorb alpha particles. Low background in beta and gamma detectors cannot be obtained by these methods because the specific ionization is not always distinguishable from that of the cosmic radiation [197]. The fact that backgrounds for unshielded alpha counters can be made by factor of 10 to 100 times smaller than fully shielded beta or gamma counters demonstrates the effectiveness of the discrimination. Therefore, massive shielding and anticoincidence systems are not considered essential to alpha counting.

Scintillation Detector

A number of organic and inorganic substances have found wide use as scintillators. Inorganic substances like ZnS and NaI are excellent phosphors in the presence of small amount of an activator like silver and thallium respectively. While, NaI (Tl) phosphor is useful in gamma spectrometry, ZnS (Ag) phosphor is specific for alpha radiation. In the present study, the alpha counting system (ACS 4004), manufactured by Electronic Corporation of India Limited) with Alpha Probe SP647A was used to measure the alpha activity in the environmental samples. The block diagram of alpha counting system used is shown in Figure 3.3. The detector consists of a ZnS (Ag) phosphor screen coupled optically to a photo multiplier and housed in a tight enclosure. A drawer assembly facilities introduction of the samples (on filter paper or stainless steel planchets) into the counting position. The electronic circuitry consists of an EHT unit, preamplifier, scaler and timer. A full view of the alpha scintillation probe and counting system used in the present work is shown in Figure 3.4. Technical specifications of the alpha counting system are presented in Appendix II.
3.4. Emanometry

Good work has been made by many researchers in the estimation of $^{226}\text{Ra}$ and $^{222}\text{Rn}$ concentration in water samples using different techniques and instruments. Hideo Higuchi et al., 1984 have used the liquid scintillation counting technique and Kottrappa 1991 had reported that radium-226 and radon-222 concentration in water can be measured using electret ion chamber base radon monitor. He reported that one pCi/l of $^{226}\text{Ra}$ can be determined with an accuracy of 10% over a 10 day period. A. Durecova, 1997 and Dimova et al., 2007 have reported that determination of $^{226}\text{Ra}$ by their alpha emitting daughters using 3M’s EMPORE Radium Rad Disks and high
adsorptive uptake of MnO$_2$ resin respectively. Kohler et al., 2002 and M. Vasile et al., 2010 have compared different methods to determine $^{226}$Ra in water. The methods are $\gamma$-ray spectrometry with 186 Kev, radiochemical separation, $\beta$-$\gamma$ coincidence, $\alpha$-particle spectrometry after radiochemical separation, BaSO$_4$ precipitation, liquid scintillation counting, $^{222}$Rn daughter nuclide method and radon emanation counting in lucas cell [198-204].

Similarly, K ozłowska et al., 1999 and Hamzah et al., 2011 have estimated the radon concentration in water samples using liquid scintillation counter and gamma spectrometry. Hakl. J et al., 1995 have developed a method to determine the dissolved radon using etch track type radon monitors. Salih et al., 2000 and Machaj, 2004 have estimated the radon concentration in water samples by bubbling of air through the water [205-209].

In the present study, radon emanation technique by bubbling of air through the water followed by transferring gas to the Lucas cell was employed to estimate the $^{226}$Ra and $^{222}$Rn concentration in water due to their high accuracy and the feasibility.

### 3.4.1 Radon Bubbler

Radon bugglers are made of corning glass with airtight joints and stop cocks. Leak proof couplings are provided for degassing the samples in radium estimation as well for transferring radon gas into Lucas cells for alpha counting. Stop cocks with Teflon stem enables to handle acidic samples for leak tight storing and sampling. Ball joints (S13) are provided for coupling the Lucas cell to the bubbler for collecting radon gas. The bugglers can be mounted on a specially designed circular stand that can hold 10 bugglers as shown in Figure 3.5. A bearing coupled central rod and bubbler holding arrangements make sampling process very convenient with the system. The sketch of the radon bubbler is shown in Figure 3.6.

### 3.4.2 Programmable Alpha counting system

A scintillation detector essentially consists of a scintillator coupled to a photo multiplier. With the proper choice of the scintillator, $\alpha$, $\beta$ and $\gamma$ spectroscopy can be performed. The interaction of ionizing radiation with the scintillator causes scintillations. When the photon from the scintillator strikes the photo cathode in the photomultiplier tube, photoelectrons are liberated. These are accelerated towards dynodes system and produce a bunch of electrons and finally reach the anode, producing a current pulse. This signal is amplified and fed to a scaler.
Figure 3.5: Photographic view of the Radon Bubblers mounted on the holder
Figure 3.6: Sketch of the Radon Bubbler
In the present study, ZnS activated with Ag is used to detect alpha particles in environmental samples. ZnS(Ag) is an inorganic scintillator, it has high scintillation efficiency equivalent to that of NaI activated with Tl. The presence of an activator with the scintillator will enhance the probability of visible photon emission. As ZnS (Ag) is available only as polycrystalline powder, it has poor conversion efficiency for fast electrons, which facilitates the easy discrimination of heavy charged particles in an intense background of gamma ray [210, 211]. The full view of the programmable alpha counting system is shown in Figure 3.7. Programmable counting system having an option to connect the alpha probe or beta probe. In the present study, we have connected an alpha probe to the counting system through a connector. The photomultiplier tube and the associated electronics to detect the scintillations emitted by the alphas impinging on ZnS(Ag) scintillator. The photomultiplier assembly is designed specifically for use with a Lucas cell and employs the PM tube in an end window configuration. The PM tube is protected by an opto-coupler based protection circuit for putting off high voltage supply to PM tube when Lucas cell is decoupled from the system. The pulses produced by the PM tube are processed inside the assembly to enable them to be counted by a programmable counting. Before using the counter, the system was allowed to stabilize for three hours. The background counts of the detector was recorded every day and was found to be 0.05 cpm. The efficiency of the detector was measured using $^{241}$Am standard sources and the details are presented in Table 3.1 and Appendix III.

Figure 3.7: Photographic view of the Programmable Alpha Counting System
3.4.3 Lucas Cell

The scintillation cell is a special device used for gas collection and estimation of radium and radon by emanometry. It was introduced by Van Dilla and modified by Lucas. It is named as Lucas cell. Raghavayya developed the device used in the present study. The sketch of the scintillation cell is shown in Figure 3.8. Lucas cell is a cylindrical vessel of 150 cc volume fitted with a Swagelok connector on one side for evacuation and sampling and a glass window on the other side. Photographic view of the lucas cell is shown in Figure 3.9. The wall of the cell is coated with Zinc Sulphide (Ag) activated scintillator which produces scintillations when impinged upon by alphas emitted by radon and its progeny. These scintillations pass through the glass window and fall on the photocathode of a photomultiplier when coupled to photomultiplier assembly. The cell can be used for repeated sampling by flushing it.

![Figure 3.8: Sketch of the Lucas cell](image)

**Table 3.1: Calibration details of alpha counting system**

<table>
<thead>
<tr>
<th>Source</th>
<th>Am-241</th>
</tr>
</thead>
<tbody>
<tr>
<td>Date of preparation</td>
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</tr>
<tr>
<td>Counts</td>
<td>4140 dpm</td>
</tr>
<tr>
<td>Activity</td>
<td>69 Bq</td>
</tr>
<tr>
<td>Operating voltage</td>
<td>700V</td>
</tr>
<tr>
<td>Bias voltage</td>
<td>800mV</td>
</tr>
<tr>
<td>Gain/Amplification</td>
<td>1</td>
</tr>
<tr>
<td>Measured background</td>
<td>0.5 cpm</td>
</tr>
<tr>
<td>Measured efficiency</td>
<td>74 %</td>
</tr>
</tbody>
</table>
Figure 3.9: Photographic view of the Lucas cell
3.5. Laser Fluorimeter

Uranium concentration in most surface and near surface continental waters is lower ranging from < 0.1 ppb to more than 1000 ppb [212]. In natural waters, however, high uranium concentrations generally have been found only in aquifers containing uranium mineralization. Different methods for uranium determination described in the literature, can be divided into two groups: “in situ” and laboratory methods. At first, each sample was measured for total alpha and beta activity. The data obtained helps us for the choice of the method to be used. Taking into account their precision, the laboratory methods includes pre-concentration through vaporization; acidification; co-precipitation; calcification; acid or carbonate separation; extraction and preparation for measuring uranium depends on the source of uranium, the geological and hydro-chemical parameters. The initial pH was always being controlled.

Number of instruments are available for the estimation of uranium concentration, some of them lack accuracy in the determination of trace amounts of uranium in aqueous samples, like particle track etch technique and fluorocolorimetry techniques [213, 214]. McDowell 1974 and Sanchez 1998 have reported a low-background liquid scintillation system to determine uranium activities in natural water samples. Instrument was operated in alpha beta separation mode and calibration curve was employed to measure the uranium content with the detection limit of 0.033 Bq/l [215, 216]. Richter et al., 1999 have measured uranium concentration in water samples using high purity germanium crystal (HpGe, 41% relative efficiency) gamma spectrometry [217]. Madrakian et al., 2007 have determined the uranium (VI) in water samples using spectrophotometry [218]. Mehra et al., 2007 have reported the fission track registration technique for the analysis of uranium concentration in water samples [77]. As per the ASTM standard procedure, uranium concentration was estimated by alpha spectrometry by many researchers [219]. Rani et al., 2013 have measured the uranium concentration in drinking water samples collected from Rajasthan using inductively coupled plasma mass spectrometry [90].
Jung et al 1987 have reported a sensitive, direct method for the determination of trace amounts of uranium in water utilizing laser-induced fluorescence spectroscopy adding a fluorescence-enhancing reagent 'Fluran'. Kutahykali et al., 2011 have compared different fluorescence enhancing reagents to measure the uranium in aqueous solution [220, 221]. Therefore, in the present investigation high sensitive Laser fluorimetry is used to estimate very low concentrations of uranium in water samples collected from open-well, lake, bore well and river water sources.

**Figure 3.10: Block diagram of laser fluorimeter**

**Principle and working of Laser Fluorimeter**

The laser based instrumental techniques, such as, fluorimeter, spectrofluorimeter, phosphorimeter were evolved during the past three decades. They utilize the half-life of phosphorescent uranyl compounds and its decay as a diagnostic method for the direct determination of uranium at \(\mu\)g\(\text{L}^{-1}\) levels in natural water samples for hydro-geochemical investigation.

When laser light absorbed by the system is not sufficiently energetic to eject the electrons from the atoms, the electrons will rise to higher energy levels. Since the lifetime in higher energy level is very less, they will come back to ground level with the emission of light energy. When the emission of light is instantaneous, the phenomenon is called fluorescence. The use of fluorescence in quantitative analysis is based on the relationships between the intensity of fluorescence and concentration of element under study and it follows Beer-Lambert’s law. The following relation gives the intensity of fluorescence.
\[ F = \Phi I_o a c l \]

Where $F =$ Intensity of fluorescence  
$I_o =$ Intensity of incident light  
$\Phi =$ Quantum yield $=$ ratio of photons emitted to the photons absorbed  
$a =$ Extinction coefficient  
$c =$ Concentration of the element present in sample and  
$l =$ Length of optical path  

For a given fluorescent compound, solvent temperature and a cell of definite dimensions,  

\[ F = k c \]

Where $k$ is the proportionality constant $= \Phi I_o a l$

**Theory of operation**

The schematic diagram of the instrumental setup is shown in Figure 3.10. Laser fluorimeter used in the present study is fabricated by Quantalase Pvt. Ltd, Indore and the know how was adopted from Raja Ramanna Centre for Advanced Technology. The instrument consists of a sealed-off molecular nitrogen laser tube as the excitation source, lasing at 337.1 nm, along with a sample compartment and a photomultiplier tube (PMT), as a radiation transducer. The detector is present at right angle to the radiation source, thus the background signal due to the source will be negligible and a lower detection level can be achieved. The laser source emits an intense pulsed ultraviolet radiation carrying an energy of 20 mJ having pulse width of 7 ns at a repetition rate of 10 pulses s\(^{-1}\). The UV beam of the laser enters the detector assembly through a low pass 350 nm cut off filter. A part of this beam (8\%) is used for monitoring laser power by a phototube and the rest falls on the sample cuvette. The 337.1 nm output of the laser is focused onto the sample cell made up of suprasil quartz (3 ml for laser fluorimeter and 7 ml for LED fluorimeter) that excites the uranium complex compounds. These compounds are formed when sodium pyrophosphate (fluorescence-enhancing agent) is added to the sample solution they may contain organic matter. The fluorescent light is detected at right angles to the incident light by a gated photomultiplier tube (PMT) through a high pass 450 nm yellow filter. During the direct fluorimetric analysis of uranium in water samples, one may get significant interference from dissolved organic compounds in the water samples. This interference can be reduced by measuring the delayed fluorescence signal in view of utilizing the difference in lifetimes of uranyl complex (200 ms) and
that of organic compounds (100 ns). Thus, the fluorescence due to organic compounds is eliminated and only the fluorescence of uranium isotopes is collected by the time-gated PMT. The fluorescence is proportional to the concentration of uranium in the aqueous medium. In addition, the fluorescence peak of organic matters is around 400 nm and that of uranium in complexing media are 490 nm to 540 nm with three characteristic peaks. Therefore, using an optical filter at 450 nm, the contribution of organic matter is eliminated [222].

**Description of the system**

The techniques comprises the steps of exciting the uranyl ion in the sample by projecting electromagnetic energy of suitable wavelengths and measure the decay with time of the uranium phosphorescence after the termination of the incident electromagnetic radiation. The signals from the phototube and photomultiplier tube are processed in the processor module. The system consists of four types of modules, namely laser power supply module, laser module, detector module and processor module.

1. Laser power supply module: This consists of a switch mode power supply, which works in resonant mode. A 30 V dc is switched on by two bipolar transistors at 25 kHz. This is stepped up by a ferrite core transformer to a high voltage. A fast recovering rectifier bridge rectifies the 25 kHz sine wave to 10 kV dc. This is then fed to storage capacitors.

2. Laser module: This module consists of laser assembly, high voltage ferrite core transformer and rectifier.

   a) Laser assembly: The laser assembly consists of two stainless steel electrodes mounted on an insulating block of metallon. Low inductance capacitors of 8 nf (30 kV) and 4.8 nf (30 kV) are mounted close to the electrodes and serve as ‘storage’ and ‘peaking’, respectively. A spark gap is used as a high voltage switch. Two vacuum connectors are provided to connect the laser cavity to vacuum pump for evacuation and to nitrogen cylinder for filling the gas. A 90° quartz prism is used for bending the laser beam out of the module.

   b) Spark gap: This consists of two electrodes of tungsten impregnated with barium and is mounted on a metalized ceramic tube. Argon at high pressure is filled in before sealing. This is used to switch the high voltage at a fast rate (ns) end to carry very high peak currents (kilo amps).
3. Detector module: The laser beam from the laser module enters the detector module through a low pass 350 nm cut off filter before splitting. A small portion of the beam (8%) passes through a neutral density (N.D) filter and enters the phototube. The output of this is proportional to the laser power and is available for processing. The remaining portion of the beam falls on the sample cell, which emits the light at right angles to the direction of the incident light. Then, the emitted light passes through a 450 nm yellow filter and falls on a photocathode of the photomultiplier tube. The photomultiplier tube amplifies the small current generated in the photocathode to a factor of about $10^6$ by the dynode chain.

4. Processor module: The output from the photomultiplier tube is then fed to the processor module. This unit analyses the pulses and finally displays the number of counts in six digits through seven segments LEDs. Finally, from the observed number of counts the concentration of uranium present in the sample was calculated.

Using the above accurate and high sensitive instrument, the natural uranium level in different environmental samples has been estimated. The photographic view of the Laser Fluorimeter is shown in Figure 3.11 and technical specifications of laser fluorimeter is shown in Appendix IV.

**Importance of Fluorescent agent**

The primary function of the fluorescence-enhancing agent (complexing agent + buffer) is the formation of the single phosphorescent uranyl species, but it also acts as a strong complexing agent for other metals in solution to reduce their effects on uranyl luminescence.

(1) The half-life of phosphorescence of uranium in an aqueous solution increases very rapidly as the concentration decreases.

(2) At low uranium concentrations, the effect of adding certain anions, notably polyphosphates to the sample is to increase the initial quantum yield. At the same time there is no enhancement of organic luminescence with the addition of these anions. Hence, thus such addition can be used to enhance the uranium luminescence selectively.

A phosphate taken from the group of pyrophosphate, tripolyphosphate, tetraphosphate, trimetaphosphate, tetrametaphosphate and hexametaphosphate is effective. This family of reagent is sensitive to acid and will decompose eventually to simple monophosphates in highly acidic solutions. The monophosphate solutions so generated are considerably less effective in stimulating the luminescent efficiency of the uranyl ion. A buffer is therefore added to reduce the acidity of the solution, since the phosphorescence enhancement is pH-dependent. The polyphosphate was added to
buffer in the ratio of about 1 to 10. The pH of the polyphosphate (pyrophosphate) solution was maintained at the optimum value for phosphorescent intensity at pH~8 by adding a phosphate buffer. This condition helps for optimum masking efficiency due to other interfering metals.

In laser fluorimetry, the choice of the fluorescence-enhancing reagent depends on the matrix composition (presence of quenching and/or absorbing species) and uranium concentration level. Kochan and Shuktomova, 1994; Premadas and Saravanakumar, 2005 have used laser fluorimetry for the direct determination of uranium in water using sodium pyrophosphate as fluorescence enhancing agent after calibrating the instrument using a standard solution [223, 224]. Rathore et al., 2001 have studied acidic buffer mixtures containing different dihydrogen phosphates plus phosphoric acid as a uranyl fluorescence-enhancing reagents in laser fluorimetry [225].

Many researchers like Campen and Bachmann, 1979; Harms et al., 1981; Kochan and Shuktomova, 1994; McHugh, 1982; Tikoo and Murty, 1980; Zook et al., 1981 have developed methods for the determination of uranium in matrices like rocks, soils and sediments, mineralized rocks and other uranium rich materials [223, 226-230]. They adopted elimination of matrix effects by a one-step or two-step dilution of sample solution. The dilution with distilled water removes matrix effects to such an extent, that quenching by impurities no longer influences the analysis.
3.6.   **LED Fluorimeter**

LED Fluorimeter is an instrument for measurement of trace amount of uranium in aqueous samples, such as water drawn from different sources. Photographic view of the LED fluorimeter is shown in Figure 3.12. The instrument works on the principal of measurement of fluorescence of uranium complexes in the aqueous sample. On excitation of uranium complexes with UV light or suitable wavelength, the uranium complexes emit green fluorescence. This can be measured by a sensitive Photomultiplier tube (PMT). Since the fluorescence yield is proportional to the intensity of excitation source and concentration of uranium in the sample, measurement of fluorescence will give information about concentration of uranium in the sample. Since the fluorescence yield varies for different complexes of uranium an inorganic reagent Fluran (Fluorescence Enhancing agent) is used to convert various complexes into single form having same fluoresce yield. Most of the organic species present in natural water fluorescence mainly in blue green region when excited by LED. This fluorescence can be blocked partially by using an optical filter allowing light only of wavelength above 475 nm. However, this does not completely eliminate the fluorescence from the organic matter. Under pulsed excitation, the fluorescence from the organic matter lasts only for few tens of nanosecond, whereas the uranium complexes emit fluorescence for a few tens of microseconds. By proper time gating of PMT, the remaining fluorescence from organic matter can be completely eliminated. By this technique concentration down to 0.2 ppb can be measured. Comparison between the Nitrogen laser and LED based Fluorimeters is tabulated in Table 3.2 and technical specifications of LED fluorimeter is shown in Appendix V.
Figure 3.12: Photographic view of the LED Fluorimeter

Description of the instrument

a) A Cuvette chamber, where the cuvette with sample is inserted.

b) A bank of intense pulsed UV LEDs, which excite fluorescence in uranium complexes in the sample. A suitable sharp cut off filter transmits only light of wavelengths shorter than 400 nm from the LEDs. Lenses appropriately arranged to focus the LED light on the sample in the cuvette.

c) Detection chamber houses the photomultiplier tube and suitable filters which transmit the fluorescence, but do not transmit the LED light. The photomultiplier tube is kept in off mode when the LEDs are on and is automatically switched on after a delay for a period of about 50 microseconds.

d) The electronics perform the following functions.
   1. Precision timing circuits for producing precisely timed pulses in the LEDs.
   2. PMT pulser to switch on the photomultiplier tube at the required time for the required duration.
   3. Low drift precision Op Amps to process the photomultiplier tube output and LED current monitor output.
   4. A Microprocessor based system acquires and process the data. The processing includes averaging of the signals over a large number of pulses, upto 2000 pulse, and applying formulae for calculating concentration of uranium from measured fluorescence. The microprocessor also permits recording the temperature of the instrument and date of measurement.
Table 3.2: Comparison between the Nitrogen laser and LED based Fluorimeters

<table>
<thead>
<tr>
<th>Sl. No.</th>
<th>Parameter</th>
<th>Nitrogen Laser based Fluorimeter</th>
<th>LED based Fluorimeter</th>
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</thead>
<tbody>
<tr>
<td>1</td>
<td>Source of excitation</td>
<td>Nitrogen gas laser</td>
<td>Light Emitting Diode</td>
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<tr>
<td>2</td>
<td>Radiation power</td>
<td>20 – 25 μJ/pulse</td>
<td>20 μJ/pulse</td>
</tr>
<tr>
<td>3</td>
<td>Repetition rate</td>
<td>10 pulse per second</td>
<td>1000 pulse per second</td>
</tr>
<tr>
<td>4</td>
<td>Pulse to pulse variation</td>
<td>~10%</td>
<td>&lt;1%</td>
</tr>
<tr>
<td>5</td>
<td>Radiation source lifetime</td>
<td>~1 year</td>
<td>~ 5 year</td>
</tr>
<tr>
<td>6</td>
<td>Fluorescence averaged over</td>
<td>20 pulses</td>
<td>2000 pulses</td>
</tr>
<tr>
<td>7</td>
<td>Analytical range</td>
<td>0.2 – 20 ppb</td>
<td>0.2 – 120 ppb</td>
</tr>
<tr>
<td>8</td>
<td>Accuracy</td>
<td>&lt;10% R.E</td>
<td>&lt; 10% R.E</td>
</tr>
<tr>
<td>9</td>
<td>Precision</td>
<td>10% S.D</td>
<td>5% S.D</td>
</tr>
<tr>
<td>10</td>
<td>Organic matter interference</td>
<td>Using optical filter and time gated</td>
<td>Using optical filter and</td>
</tr>
<tr>
<td></td>
<td>removal</td>
<td>measurements</td>
<td>time gated measurements</td>
</tr>
<tr>
<td>11</td>
<td>Sample volume for analysis</td>
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<td>5.5 ml (5 ml sample + 0.5 ml Fluran at pH 7)</td>
</tr>
<tr>
<td>12</td>
<td>Fluorescence enhancing agent</td>
<td>Buffer of sodium phosphate (5%) and</td>
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<tr>
<td></td>
<td></td>
<td>phosphoric acid (pH-7)</td>
<td>phosphoric acid (pH-7)</td>
</tr>
<tr>
<td>13</td>
<td>Quantitative analysis</td>
<td>Standard addition method</td>
<td>Standard addition method</td>
</tr>
</tbody>
</table>

3.7. Gamma Spectrometry

Gamma Spectrometry is the method of qualitative and quantitative estimation of radioisotopes by measuring the energy distribution of the gamma radiations emitted by the radioactive isotopes. A spectrometer is an instrument that separates the output pulses from detectors, according to height of the pulses. Since the pulse height is proportional to the energy of the radiation, the output of the spectrometer provides detailed information, that is useful in identifying the unknown radioisotopes and in the determination of the radioactivity of one isotope in the presence of others.

Gamma spectrometry uses either NaI (TI) detector or semiconductor detectors like Ge(Li) or Si(Li). Spectrometry using solid-state detectors has brought marked improvement in many fields such as nuclear spectroscopy, nuclear fission research, activation analysis, environmental studies, analysis of food etc. Primarily, owing to their inherent quality, Si(Li), Ge(Li) and Hyper Pure Germanium (HPGe) detectors having better resolution, enable us to distinguish between closely spaced energies. One advantage of such direct and non-destructive analysis is that chemical separation techniques can be avoided or kept to a minimum in most of the routine analysis work. Hence, it provides an easy and rapid method for measurement of natural radioactivity in the environmental samples.
Hyper Purity Germanium (HPGe) Detectors:

The HPGe detectors possess all the useful characteristics of Ge (Li) detectors and in addition to that, they have the advantage that, they can be stored at room temperature. Cooling with liquid nitrogen is required only when the detector is in use. In the present investigation, an HPGe spectrometer was used to measure the concentrations of gamma emitting radionuclides in soil and rock samples. The HPGe gamma ray detector essentially consists of detector element and cryostat-Dewar and electronic components of the analysis system. HPGe detector used in the present study was fabricated by EG&G ORTEP, USA with a crystal dimension of $47.9 \times 52.0$ mm. The photographic view of the HPGe detector is shown in Figure 3.13.

Detector Element:

The semiconductor detector element is a single crystal of high purity germanium made into a diode capable of withstanding high reverse bias voltage, at cryogenic
temperature. The purpose of maintaining the detector at liquid nitrogen temperature is to lower the leakage current and hence reduce the noise of the system. At cryogenic temperature the electron hole pairs produced by the absorption of an X-ray or gamma ray photon are swept to opposite contacts by an electric field. The resulting induced current pulse is integrated by a charge sensitive preamplifier producing an output voltage pulse with its height proportional to the incident gamma ray energy. The relevant specifications of Gamma-X series p-type semiconductor detector is given in Appendix VI.

**Cryostat Dewar Configuration:**

The cryostat-Dewar system maintains the detector element in a high vacuum at close to liquid nitrogen temperature. The Dewar serves as a reservoir of liquid nitrogen, while the cryostat provides a path for heat transfer from the detector element to the liquid nitrogen reservoir. Both the Dewar and the cryostat relay upon a vacuum to insulate cold inner parts from the outer surface.

The cryostat provides an outer envelope, which can be maintained at high vacuum; In addition, the cryostat contains a mount for the detector element and associated electronic components. Vacuum feed through the cryostat allows electrical connections to be made from the detector to the preamplifier and high voltage filter. For the purpose of the present study, the detector assembly was housed inside a shield of length 50 cm consisting of 10 cm thick lead plate with aluminum lining. A free gap of 20 cm is provided between the detector end cap and the shielding material in order to keep the samples and to minimize the back scattering of gamma rays.

**Electronic Components of the Analysis System:**

The associated electronic system required for data acquisition and processing consists of a low noise charge sensitive preamplifier, a high voltage filter, a linear amplifier, high voltage bias supply, pulse height analyzer, analog to digital converter and multichannel analyzer. The spectrometer is provided with computer software for the operation and spectral analysis.