CHAPTER III

Experimental Set Up

A. Description of Various Components of the Experimental Assembly

1. Radioactive Sources for Calibration of the Gamma Ray Spectrometer
2. Weak Beta Source for Production of External Bremsstrahlung
3. Beta to Gamma Converter Foil
4. Perspex Absorber
5. ORTEC HPGe Detector
6. Auxiliary Electronics

B. Standardization and Calibration of Gamma Ray Spectrometer
A. Description of various components of the experimental assembly.

The experimental assembly adopted for the present investigations consists of a weak beta source, converter foil, perspex absorber, target and a high-energy resolution HPGe gamma ray spectrometer. In the following Section we describe in detail various parts of the experimental assembly. The high-energy resolution HPGe detector has been calibrated using many gamma sources.

1. Radioactive sources for calibration of the gamma ray spectrometer:

The HPGe detector-spectrometer used in the present study is calibrated by adopting the standard method before each set of data is acquired. Similarly each acquisition is followed by the verification of the calibration to confirm the stability of the detector and MCA. The spectrometer calibration involves the selection of a set of monoenergetic gamma sources. The energy, half-life and strength of these sources are presented in Table 3.1. These sources are procured from Board of Radiation and Isotope Technology (BRIT), Mumbai, India. We checked the purity of each source by scanning the gamma and X ray spectra using $\frac{3}{4} \times 2^\prime$ NaI(Tl) scintillation detector coupled to MCA before using them for calibration of HPGe detector spectrometer. It is confirmed that no unexpected gamma or X ray lines are present in the emissions from these radioactive sources. We wanted the detector spectrometer to be calibrated in the energy range from 10 keV to 150 keV and the sources are selected accordingly to provide monoenergetic peaks in this energy range.

$^{57}$Co radioactive source:

The radioactive $^{57}$Co nucleus decays purely by electron capture to the excited state of $^{57}$Fe nucleus (136.473 keV) and subsequently to the ground state. The excited state of $^{57}$Fe nucleus emits gamma rays of energy 122.061 keV.
(85%) and 136.473 keV (11%) and 14.411 keV (8.5%). Because of internal conversion process the Fe K X ray photons such as Kα₁, Kα₂ and Kβ₁ are also emitted. The energies of these X rays are 6.404, 6.391 and 7.058 keV respectively (E. Browne et al. 1986). From this source, the gamma rays of energy 136.473 keV, 122.061 keV and 14.411 keV are used for calibration of the gamma ray spectrometer.

\textbf{109\textsuperscript{Cd} radioactive source:}

The radioactive \textsuperscript{109}Cd nucleus decays purely by electron capture to the excited state of \textsuperscript{109}Ag. The excited state of \textsuperscript{109}Ag goes to the ground state of \textsuperscript{109}Ag by emitting 88.034 keV gamma rays. The spectrum of \textsuperscript{109}Cd nucleus therefore contains two major lines; one corresponding to 88.034 keV and the other corresponding to \textsuperscript{109}Ag X ray of energy 22.163 keV. The gamma energy of 88.034 keV is used for the calibration of the gamma ray spectrometer.

\textbf{241\textsuperscript{Am} radioactive source:}

The radioactive \textsuperscript{241}Am nucleus decays to the various excited levels of \textsuperscript{237}Np purely through α- emission. The excited \textsuperscript{237}Np predominantly emits two gamma rays of energies 59.536 keV and 26.344 keV. The gamma energy 59.536 keV is used for calibration of the gamma ray spectrometer.

The decay schemes of these sources used for the calibration of the gamma ray spectrometer are illustrated schematically in figures 3.1, 3.2 and 3.3.
Figure 3.1: Decay scheme of $^{57}$Co

Figure 3.2: Decay scheme of $^{109}$Cd
Figure 3.3: Decay scheme of $^{241}$Am
Table 3.1: Description of the sources used for the calibration of gamma ray spectrometer.

<table>
<thead>
<tr>
<th>Name of the source</th>
<th>Half life</th>
<th>Strength (Bq)</th>
<th>Gamma energies (keV)</th>
<th>Intensity (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>$^{57}$Co</td>
<td>272 days</td>
<td>~ 100</td>
<td>136.473</td>
<td>10.69</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>122.061</td>
<td>85.50</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>14.411</td>
<td>9.54</td>
</tr>
<tr>
<td>$^{109}$Cd</td>
<td>453 days</td>
<td>3700</td>
<td>88.034</td>
<td>3.70</td>
</tr>
<tr>
<td>$^{241}$Am</td>
<td>433 years</td>
<td>740</td>
<td>59.536</td>
<td>36.00</td>
</tr>
</tbody>
</table>

2. Weak β source for production of external bremsstrahlung [EB]:

The external bremsstrahlung having continuous energy spectrum is produced when charged particles interact with the Coulomb field of the nuclei in the target. It is well known that the intensity of EB photons, in a thin target, is proportional to square of the atomic number of the target and inversely proportional to mass of the incident particle. Because of these properties, beta particles can produce more intense EB photons in high Z targets. We have used a $^{90}$Sr-$^{90}$Y beta source which emits 0.5462 and 2.281 MeV beta particles. The half-life of $^{90}$Sr is 28.4 years and that of $^{90}$Y is 64 hours. Therefore $^{90}$Y is in secular equilibrium with $^{90}$Sr. Because of long half-life of $^{90}$Sr, the intensity of beta particles remains constant throughout the experiment. The decay scheme of $^{90}$Sr-$^{90}$Y radioactive nuclei is shown in Figure 3.4.

The $^{90}$Sr-$^{90}$Y source is prepared by dissolving Sr (NO$_3$)$_2$ in HNO$_3$ solution and then it is deposited on a thin mylar foil. After drying the source with infrared lamp it is covered with a thin mylar foil and sandwiched between two plastic discs of thickness 5 mm and diameter 25 mm with an annular opening of 10 mm diameter on one side. The schematic diagram of beta source with collimation arrangement is shown in Figure 3.5.
Figure 3.4: Decay scheme of $^{90}$Sr-$^{90}$Y beta source

Figure 3.5: Collimation of $^{90}$Sr-$^{90}$Y beta source: S-source, P-perspex
3. **Beta to gamma converter foil:**

In this experiment we use a converter to convert the beta particles into EB photons. As mentioned earlier the EB intensity is maximum for high Z elements. But we have selected nickel (Z=28) as a converter because the K X ray peaks of nickel do not come in the way of K absorption edge of the targets used in the present investigations. The thickness of nickel converter is selected to produce maximum intensity of EB photons in the energy region from 25 keV to 100 keV because the K shell binding energies of the targets selected in the present study lie between 50-90 keV. We also scanned EB spectrum using Cd and Cu as converters and found that the EB spectrum was almost the same with a small shift in peak energy.

4. **Perspex absorber:**

The beta particles from $^{90}$Sr-$^{90}$Y are used to produce EB photons in the converter. These EB photons are allowed to pass through the target whose K shell photoelectric parameters were determined. Since we used the nickel foil (47.2 mg/cm$^2$) to produce EB photons, some beta particles from the source undoubtedly pass through the converter without producing EB photons. These transmitted beta particles may produce EB photons in the target and also in the detector. Presence of these unwanted EB photons would distort the measured spectrum. In order to prevent these beta particles from reaching the target and the detector, a perspex absorber of thickness 10 mm is placed between the nickel converter and target. As perspex is a low Z material (effective Z ~ 5.8), it can stop all the beta particles transmitted through the converter with minimum production of EB photons in it.

5. **ORTEC HPGe detector:**

In the present study we used an ORTEC HPGe detector of type GMX 10P for recording the spectrum of EB photons transmitted through the target. It is a High Purity Germanium (HPGe) detector that is be used for measuring both Gamma (GM) and X ray (X) photons. The warranted relative efficiency of the
detector is 10%. The detector capsule is mounted horizontally over the cryostat rod. Such a mounting is called pop-top (P) mounting. The detector can be employed to measure the X ray and gamma photons of energy in the range from 3 keV to 10 MeV.

ORTEC HPGe crystals are grown in their laboratory using Czochralski technique. A germanium-seed crystal is dipped into molten germanium and slowly withdrawn rotating it in hydrogen atmosphere so as to harbor the growth of new single cylindrical Ge crystal at its tip. The rate of rotation and withdrawal of the seed crystal as well as temperature of the melt are adjusted to control the growth of the crystal. The level of impurities in the crystal is assessed by Hall effect measurements to classify it into p or n type. Only crystals of adequate purity and crystallographic perfection are later mechanically sliced to suitable size. They are drilled partially along the axis for insertion of electrical contact-pin and then polished to a perfect cylindrical shape. The bulleted shape given to the crystal in the front would produce signals of superior peak shape and timing characteristics. A neatly labeled schematic diagram of n type detector element of our HPGe is shown in Figure 3.6. It has 600-micron thick lithium drifted layer on the surface of the drilled cross section and interior of the hole for good electrical contact. The P+ contact was obtained on the surface by implanting a thin sputtered boron ion layer of 0.3 micron. A protection cover of thickness 0.13 mm made of aluminized mylar was laid on the cylindrical surface. The crystal of germanium is 49.6 mm in diameter and length 47.1 mm with an inactive germanium of 0.3 μm. It was mounted in a hollow cylindrical mounting-cup with the help of screws.

The Pop Top detector capsule is schematically shown in Figure 3.7. The front of the capsule was covered by a beryllium window of 0.5 mm (19 mil) thickness with a gap of 3 mm from the detector crystal. The high order vacuum inside the detector capsule would protect the crystal from temperature shock and oxidation. The 'boron-ion implanted-contact' and the beryllium window together allow photons of energy down to 3 keV to enter the active volume of
the detector. The germanium K absorption edge is 11 keV; except photons of 11 keV, this detector detected the photons of energy up to 10 MeV. The pop-top detector capsule was parted into vacuum and non-vacuum compartments. The former was a sealed and self-pumping cryogenic enclosure that contained the HPGe detector element positioned coaxially over a central contact pin followed by front-end electronics.

![Diagram of ORTEC-HPGe (n-type) detector element.](image)

**Figure 3.6:** ORTEC-HPGe (n-type) detector element.

The front-end electronics consisted of a cooled FET as temperature sensing device and a resistor of junction transistor that formed the first stage of preamplifier. This unit provided either resistive feedback or 'transistor reset' active feedback of the necessary pre-cooling attained in the detector element. The second stage of preamplifier and the high voltage (HV) filter were also part of the detector capsule housed in the non-vacuum compartment. The liquid nitrogen (LN$_2$) cooled cryostat provided an initial cooling of the detector material to about -196 °C (77K). The thermal connection between dipstick and the detector mounting-cup was made with aluminium, which acted as a good
thermal conductor and electrical resistor. The detector cup must be maintained at 85-100K.

This pre-cooling is very essential for the safety and efficient functioning of the detector without any thermal noise. The electron mobility is increased by 10 times and hole mobility by 20 times at 77K compared to the values at 300K. The carrier saturation velocity is almost doubled at 77K compared to the value at 300K. The energy utilization for production of each hole-electron pair also considerably reduced to 2.96 eV at 77K. After necessary pre-cooling, we applied the bias voltage in the steps described in part B. The performance features of the detector are shown in Table 3.2.

Figure 3.7: Schematic diagram of ORTEC -HPGe detector capsule; A- vacuum and B- non-vacuum compartments
Table 3.2: Performance features of the HPGe detector of type GMX 10P.

<table>
<thead>
<tr>
<th>Item</th>
<th>Energy Source</th>
<th>Measured magnitude</th>
<th>Amplifier Time-constant</th>
</tr>
</thead>
<tbody>
<tr>
<td>Resolution (FWHM)</td>
<td>1.33 MeV</td>
<td>40Co</td>
<td>1.77 keV</td>
</tr>
<tr>
<td>Peak to Compton ratio</td>
<td>---</td>
<td>40Co</td>
<td>50:1</td>
</tr>
<tr>
<td>Relative Efficiency</td>
<td>1.33 MeV</td>
<td>40Co</td>
<td>19%</td>
</tr>
<tr>
<td>Peak Shape (FWTM/FWHM)</td>
<td>---</td>
<td>40Co</td>
<td>1.9</td>
</tr>
<tr>
<td>Resolution (FWHM)</td>
<td>5.90 keV</td>
<td>55Fe</td>
<td>600 eV</td>
</tr>
</tbody>
</table>

*FWHM-full width at half maximum, FWTM-full width at tenth maximum

The block diagram of gamma ray spectrometer assembly with ORTEC-HPGe detector is shown in Figure 3.8.

![Block diagram of ORTEC-HPGe detector spectrometer assembly.](image)

Figure 3.8: Block diagram of ORTEC-HPGe detector spectrometer assembly.
• Cryostat:

The cryostat of the present HPGe detector is 'dip-stick' dewar model. The dewar is of capacity 30 liters of LN$_2$. The dipstick has a RTC silicon-rubber collar that formed a gas tight seal. The silicon collar contained two stainless steel tubes, which were used for LN$_2$ filling and vapor exhaust. These tubes extended up to 6" deep into dewar neck. In the case of leakage of LN$_2$ at the collar these tubes prevented the liquid level from rising within 6" from dewar neck. We filled LN$_2$ in the cryostat always as per the manufacturers instructions.

5. Auxiliary electronics:

The detector assembly consists of a set of active electronic modules like high voltage unit, HV filter, charge sensitive preamplifier, spectroscopic coarse gain and fine gain amplifier, 8K multichannel analyzer and computer interface. These modules are broadly classified as electronics inside the detector capsule and the electronics outside the capsule. Front-end electronics and HV filter with preamplifier form the electronics within the capsule. A high voltage unit, linear spectroscopic amplifier and multichannel analyzer (MCA) together form the electronics outside the capsule.

a. Electronics within the detector capsule:

Electronics within the HPGe detector capsule both in vacuum and non-vacuum compartments are located on cryogenic rod to benefit from the low temperature that prevails inside the capsule for better performance without noise.

1) Front-end electronics:

The electronics housed inside cryogenically cooled front end of HPGe detector capsule forms the front-end electronics. It includes FET and a feedback element of the charge sensitive preamplifier that acts as an electronic
switch to reset the input field-effect transistor (FET) gate voltage to zero whenever the given voltage reaches the threshold. The feedback element comprises a capacitor and a resistor or a transistor either to form a passive or active feedback respectively. The active feedback provides ultrahigh count rates. The temperature-sensing element (TSE) will automatically shut off the HV power supply if the detector temperature begins to rise. The whole arrangement of front-end electronics along with cryogenic enclosure reduces electronic noise as both the input FET and feedback element have optimum noise characteristics at 120K.

ii) HV filter and Preamplifier:

Both HV filter and preamplifier units are sealed and located behind the detector element within the pop-top capsule. They are outside the vacuum chamber but mounted on the same cryogenic rod to stay cooler than room temperature. The HV filter model number 138 (SPN12288) has a very long time-constant. This ensures the application of the high negative bias (-4000V) to the detector in gradual steps, which protects the FET of front-end electronics from voltage spikes and from damages occurring due to application of sudden full bias voltage when detector is cold and auto shutdown option is off.

The preamplifier model 257N(S/N 907) supplied with the detector is a resistive feed back amplifier, which requires only one watt of power. The amplifier is charge sensitive (175 mV/MeV) type that had front end cryogenically cooled for lowest electronic noise and highest energy resolution.

b. Electronics outside the detector capsule:

The electronics inside the detector capsule are connected to electronics outside that consists of ORTEC high voltage (HV) supply unit and ORTEC spectroscopic amplifier through BNC cables. The output of amplifier is fed to an
ORTEC multichannel analyzer (MCA) with a computer interface facility. A brief account of the special features of HV unit, amplifier and MCA are as follows.

i) High voltage unit (model HV 659):

ORTEC HV model 659 is compatible with both germanium and silicon detectors. Its design provides a high bias voltage up to 5 kV in the steps of 0-500 V at 0-100 μA current. The remote shutdown facility for non-cryogenic temperatures is the safety feature recently added to the unit by ORTEC. Output from the temperature sensors will trigger the auto shut down option. The ‘Reset’ safety switch minimises the risk of damage to the preamplifier FET under such circumstances. The selected output polarity is displayed at indicators ‘Positive’ or ‘Negative’ in the panel before the bias voltage is turned on. The ‘Overload’ indicator is an automatic overload protection embedded as an extra feature of the HV unit model 659.

ii) Premium spectroscopic amplifier (PSA 672):

This ORTEC spectroscopic amplifier of model PSA 672 is of ideal design to suit high purity germanium detector. It accepts the signals of both polarities positive and negative from the detector. The positive 0-10 V output signal provided by the amplifier is most suitable for single or multichannel pulse height analyzer. The gain is continuously variable from 2.5 to 1500. Also the shaping time constants are adjustable from 0.5, 1, 2, 3, 6 and 10 μS. Some more special features of the amplifier are,

- It can be used for high performance energy spectroscopy with all types of detectors.
- It has special feature called Peak Zero (PZ) adjustment in both auto and manual mode, which is compatible with any detector.
- It has pile up rejecter (PUR), with separate auto noise discriminator and % rejection LED.
• It has pulse-shaping facility in both semi-Gaussian and quasi-triangular modes. These pulse shapes can take place in either unipolar or bipolar way. Proper choice of these pulse-shaping filters effectively doubled the time constants available for optimum resolution.

• Automatic base line restorer (BLR) helps automatically in noise discrimination. This leads to superior performance at both low and high counting rates.

• It has different input provision for reduction of ground loop noise.

• It has built in 'over load protection' for pulse reset amplifiers, which automatically compensates for reset recovery.

iii) Multichannel analyzer (MCA) and associated MCA emulation software:

The ORTEC multichannel pulse height analyzer (MCA) consisted of an analog to digital converter (ADC), a histogram memory and a visual display of the histogram recorded in the memory implemented in a personal computer. The digital output of ADC is proportional to the energy of the absorbed photon.

The present ORTEC MCA is a combination of specific hardware module called multichannel buffer (MCB) and MCA emulation software called MAESTRO. The trump card can be plugged into an option slot on a personal computer to perform the action of computer controlled multichannel analyzer. The ADC specifications in the trump card are,

• Maximum ADC conversion gain is 8K with options selectable as 512, 1024, 2048, 4096 and 8192.

• ADC conversion time is 8μS. This 'dead time' loss is automatically corrected in 'live time' to show 'real time'.

• ADC is a successive-approximation (8K) type with data memory capacity of 2 billion \( (2^{31}-1) \) counts per channel.

The dual ported data memory transfers the entire spectrum from the card memory into the PC memory in milliseconds thereby allowing snapshots of the
data to be taken and processed in real time. The MCA emulation software MAESTRO-32 is a visual link that matches this ADC to provide a meaningful access to the MCB.

MAESTRO-32 is the latest version of MAESTRO with several enhancements in display options. The spectrum and the menu for actions are available on the screen together. All important operations on the spectrum such as peak location, insertions of regions of Interest (ROI) and 'display scaling & sizing' are easily implemented using both the keyboard and mouse. Spectrum peak searching, report generation, printing, archiving, calibration and other analysis tools are available from the menus. A buffer is maintained in the computer memory to which one spectrum at a time can be moved for display and analysis either from the detector memory or from disc while another spectrum was being collected in the detector. Data can be directly analyzed in the detector hardware memory. With a memory mapped MCB interface like TRUMP, the data acquisition software MAESTRO will run on any PC that supports Microsoft Windows 95, 98, 2000 and XP versions of operating systems.

B. Standardization and calibration of gamma ray spectrometer:

Before we used the HPGe detector for data acquisition we standardized the detector assembly by using standard procedure. The following are few important steps adopted for standardization.

- Sufficient level of liquid nitrogen is ensured in the cryostat and a pre-cool period of 1 hour is allowed after each $\text{LN}_2$ filling session. The laboratory temperature is maintained throughout at $22^\circ\text{C}$ with the application of an air conditioner (AC), in order to reduce the loss of $\text{LN}_2$ by evaporation.
- Initially negative bias is selected in the HV unit. The HV bias is applied every time in a step-by-step sequence. After the BIN switch is put ON the 'Reset' is pressed before the toggle switch of bias voltage is put ON. Pressing 'Reset' will put OFF the 'shutdown' LED only when the detector
element is cooled to LN$_2$ temperature. Now the toggle switch is put ON and the bias is applied by rotating the POT division by division. Each 500 volts applied is indicated by the glow of an LED in the array of 10 LEDs. When 8 LEDs in the array glow, the bias would reach -4000 volts.

- The spectroscopic amplifier is set for optimum performance in the present work by selecting the coarse gain to be 500, fine gain to be 1.05, UNI shaping at triangle position, shaping time set to 6 μS, BLR set to 'Auto' mode, 'Input' at normal -ve and 'output' set at unipolar.
- A warm up time of 1 hour is allowed for the stabilization of the electronics.
- The lid-cap of the detector window is opened to allow the gamma rays on to the detector element for the record of observations.

After initial steps of standardization, we calibrated the gamma ray spectrometer using a set of monoenergetic gamma sources by using standard method. The sources and their standard energies used for calibration of the gamma ray spectrometer are given in Table 3.1. The following are some of the important steps that we followed for calibration of the gamma ray spectrometer.

- A spectrum of each source chosen for calibration is acquired for sufficiently long period so that the counts under each peak of required energy are more than 10000.
- Marking ROI by placing the cursor symmetrically on two sides of required energy peak, the information about the channel number corresponding to the peak is obtained by clicking on 'peak information'. Destroying the past calibration, the new channel number and corresponding energy of the peak are fed to recalibrate the detector. The procedure is repeated for the selected energy peaks of already described sources.
- A calibration graph of energy versus peak channel number is plotted. The nature of calibration curve is shown in Figure 3.9. The data points lie in a straight line. A linear fit to the data points gave a straight line passing through the origin with slope calculated by default. The slope so obtained
is the required calibration constant. In our case the calibration constant is about $19.960 \pm 0.001$ eV per channel for 8K channel. The calibration constant is obtained twice for each data; once before the acquisition and next when the acquisition was over. The calibration constant is found to remain the same ensuring no drift in the channels.

The resolution of the detector spectrometer is defined as the ratio of FWHM of the photo peak to the channel number corresponding to the photo peak. We checked the detector resolution now and then for peaks of 122 keV of $^{57}$Co and 5.9 keV of $^{55}$Fe. The values were confirmed to be 700 eV and 600 eV respectively as per the specifications of the manufacturer.
References: