Chapter 3

Experimental Techniques

3.1 Introduction

In the recent years the environment involves nuclear radiation. Mankind has added to this by production of power and by application of artificial radiation to medical and other uses. With the increasing use of radioactive materials it is becoming important to know with some accuracy how the interaction of gamma rays with matter varies with gamma ray energy and atomic number [1]. In addition to usual materials a search for the other non-radioactive materials, which can be used as a shielding materials, is in progress. By determining linear attenuation coefficient, mass attenuation coefficient, of elements and other composite materials may consist of many elements, so it is very interesting to apply the Hubbell’s mixture rule for such a multi-element materials. The experimental values of linear attenuation coefficient, mass attenuation coefficient, total photon interaction cross-section, electronic cross-section and effective atomic number of the materials and their values gives us a idea about the materials which is good for the attenuation of gamma rays.

By keeping this point in the present study the absorber like lead, silver, copper, iron, tin, aluminum and composite ferrite materials which are 99.9% pure. The composite ferrite material having general formula MFe$_2$O$_4$, where M stands the divalent metallic cations (M = Co, Cu, Mg, Ni, Zn etc.) have been studied for its possible use in shielding purpose by determining its mass absorption coefficient and other related parameters.
A narrow beam geometry technique has been used to determine the linear attenuation coefficient, mass attenuation coefficient and related parameter.

3.2 Detectors

Now a day’s detectors are the important tool in radiation physics as they are playing an important role in radiation measurements. Radiations loose their energy directly or indirectly by ionization or excitation of atoms or molecules in the medium they pass through. Alpha and beta are charged particles while gamma and X-rays are neutral. There are many detectors for detecting radiations, and they are classified as 1) Detector based on ion collection method and 2) Detector based on other than ion collection method. Detector based ion collection method have been processed by two ways such as saturation collection and multiplication ion collection. Generally the saturation collections are called ionization chamber, which is occurred in gases and in semiconductor. In multiplication ion collection there are two counters 1) Proportional counter and 2) G.M counter. On the basis of detector other than ion collection have the two methods such as counter based ion light emission and image forming devices. The counters based on light emission are scintillation counter and Cerenkov counter, and image forming devices are photographic emulsion, cloud chamber, bubble chamber and spark chamber.

3.3 Selection of detector

Selection of detector are depends upon the type of radiation to be detected. We are interested in gamma ray detection and the detector should have the following characteristics,
1) It has high efficiency of detection of gamma ray.

2) Good resolution power.

3) Linear response to electron.

4) Good mechanical and electrical stability.

All the above properties are seen in the scintillation detector. For scintillation detector the scintillation material should be ideal and they posses following properties.

1) It should have very high scintillation efficiency.

2) The medium should be transparent to the wavelength of its own emission for good light collection.

3) The decay time of induced luminescence should be short so that fast signal pulses can be near that of glass (i.e. 1.5) to permit efficiency coupling of scintillation light to photomultiplier tube.

3.3.1 Types of Scintillation detector

The scintillation detectors are classified into three groups depending upon the nature of material.

1) Organic scintillation detector

2) Inorganic scintillation detector and

3) Light collection and scintillation detector mounting.

On comparing these detectors, the inorganic scintillation detectors are found to be the best detectors. For gamma ray detection the scintillation detector gives a typical output from PMT. That collects the light with a long time constant measuring circuit. The scintillation counters have the good efficiency for fast secondary electrons produced by gamma ray interaction in material.
Finally we concludes that NaI(Tl) is the best detector for gamma ray detection. CsI(Tl) is non Hygroscopic and has higher density as compared to NaI(Tl) but it is not used because for the following rasion,
1) Due to smaller forbidden gap its light yield in photons almost double than that from NaI(Tl).
2) Its decay time is nearly four times lesser than that of NaI(Tl) and thus is more suitable for higher counting rates (i.e. decay constant for NaI(Tl) and CsI(Tl) are 0.23 and 1.0 respectively).

In 1948, Robert Hofstadr first demonstrated that crystalline iodide which is activated with thallium produces an approximately large scintillation light output as compared with organic material.

The high purity of sodium iodide is activated with about $10^{-3}$ moles of fraction of thallium for growing the large Ingots. NaI(Tl) detector will damage due to water absorption if exposed to atmosphere for a small time. Crystal must be inserted in air tight container for use.

### 3.3.2 Scintillation counter

The scintillation counter is widely used for qualitative and quantitative measurements of photon radiation. The scintillation counter consist of the luminescent material known as scintillation reflecting layer such as aluminum foil enclosing the luminescent substances to facilitate the collection of light, light pipe, photomultiplier tube, amplifier, voltage discriminator and electric circuit to record the output pulses. The scintillation counter is a solid state radiation detector which uses scintillation crystals (Phosphor) to detect radiation and produce light pulses. The light pulse is converted to an electric pulse by photomultiplier
tube (PMT). The photomultiplier tube consists of photocathode, a focusing electrode and 10 dynodes that multiply the number of electrons striking at each dynode. When the photon strikes on the crystal the electrons jump from the valence band to the conduction band, leaving behind vacancy of electrons in the valence band. From the conduction band the electrons either directly jump to the valence band or via one of the intermediate levels called as traps or activator centers. The balance energy in the latter cases is emitted out as useful photon. For the present study we have used a single channel analyzer for the detection of photon (Gamma ray) due to its better stability, high quality resolution and high efficiency.

3.3.3 Multichannel analyzer

Multichannel analyzer is sophisticated device which sort out incoming pulses. According to pulse height, keep the count at each height in at multichannel memory. The counters of each channel can then be displaced on a screen or printed out to give a pulse height spectrum.

Multichannel analyzers are commonly used over a single channel analyzer, which uses a microprocessor technology with a high speed, high density, smaller size, easily computable, semiconductor memories. Now generally 4K and 8K multichannel are available in the market. The interfaces of output ports of control units are led to cathode ray tube in order to display output in terms of channel versus counts.

But we use the single channel analyzer for the present study which is supplied by nucleonix system private Ltd.
3.4 Radiation sources and energy calibration

In the present study the photon transmission measurement were done under a narrow beam geometry employing high resolution (NaI(Tl)) as a photon detector. The NaI(Tl) is an inorganic scintillation; crystals of alkali halides as NaI(Tl) are good inorganic scintillations. The energy gap between the valance band and conduction band is order of 5 – 6 eV. When the radiation energy is absorbed, the electrons from the valance band are excited to the conduction band leaving holes in the valance band. The photomultiplier tubes have good sensitivity for photons of the visible region; some of the photons reaching the photocathode of photomultiplier tube causes photoelectric effect and liberate electrons. These electrons are multiplied by a factor of about $10^6$ by series dynodes inside the PMT. The wavelength of the photons has to be shifted to visible region. In NaI(Tl) crystal detector about 30eV energy is needed to produce a photon in the visible region. In the present work radioactive sources are used to determine the linear attenuation coefficient, mass attenuation coefficient of the materials and related parameters.

The energy sources of gamma rays are used to study the photon interaction cross-section are $\text{Ba}^{133}$, $\text{Cs}^{137}$, $\text{Co}^{60}$ and $\text{Na}^{22}$. The gamma ray sources $\text{Ba}^{133}$ and $\text{Cs}^{137}$ having a single energy 0.360 MeV and 0.662 MeV but the $\text{Co}^{60}$ has the two different energies 1.17 MeV and 1.33 MeV, also $\text{Na}^{22}$ having two different energies 0.511 MeV and 1.28 MeV which is shown in table 3.1. The linear attenuation coefficient, mass attenuation coefficient and the related parameter of the elemental solids of Lead, Silver, Copper, Ferrous, Tin, Aluminium and composite spinel ferrite
materials $\text{Ni}_{0.2}\text{Zn}_{0.5}\text{Cu}_{0.3}\text{Fe}_2\text{O}_4$ are calculated under the full energy peak. Narrow beam geometry is used to calibrate the detector using single channel analyzer in the photon energy range 0.360 MeV to 1.33 MeV. The detector is calibrated using the controls of attenuator and gain of pre-amplifier. The calibration line in Fig.3.1 shows linear relationship between gamma ray energy peak channel numbers of photo peak. The selected spectrums are smoothed to avoid the statistical variation cause from random nature of radioactive decay and background counts.

### 3.4.1 Narrow beam geometry

Narrow beam geometry technique is used to measure the linear attenuation coefficient, mass attenuation coefficient and total photon interaction cross-section of the absorber like lead, silver, copper, iron, tin, aluminum and composite spinel ferrite $\text{Ni}_{0.2}\text{Zn}_{0.5}\text{Cu}_{0.3}\text{Fe}_2\text{O}_4$.

Narrow beam photon is defined by circular aperture in two or more massive shields, or collimators. When the thin uniform absorber is placed under the well collimated beam, all photons which are incoherently or coherently scattered by few degrees are prevented from reaching the detector. Such arrangements are called as narrow beam or good geometry technique [2].

### 3.4.2 Resolution of photo peak and energy calibration of gamma ray Spectrometer

The resolution of a spectrometer is a measure of its ability to resolve two peaks that are fairly close together in energy [3]. The fluctuations around the mean value are statistical in nature and lead to almost a Gaussian shape of the observed photo peak. For pulse height spectrum resolution of
energy is a ratio of the number of channel in the full width at half maximum of photo peak (FWHM) to the channel number of highest peak. For normal distribution, FWHM is equal to two times the standard deviation.

\[ R = \frac{\text{FWHM}}{\text{Pulse height of photo peak}} \times 100 \% \]  

3.1

3.5 Experimental Setup

In the present work accurate calculation of the dispersion correction, it is essential to measure the total attenuation coefficient of the elements of wide interest with good degree of accuracy over a wide range of energies. For this purpose we have measured the linear attenuation coefficient, mass attenuation coefficient and related parameter of elements Lead, Silver, Copper, Ferrous, Tin, Aluminium thin foils with high purity of order 99.9%. The purity of absorber was measured by comparing theoretical and calculated density of absorber by weighing accurately on analytical sensitive balance and circular area of thin uniform thickness 0.15 cm -1.50 cm for each additional absorber and composite material Ni_{0.3}Zn_{0.5}Cu_{0.2}Fe_{2}O_{4} ferrites in the energy range 0.360 MeV to 1.33 MeV in a narrow beam good geometry setup [4]. The essential requirements of a good counting system in the work are as follows.

- Good narrow beam collimators for incident and transmitted beam
- Incident photon beam must be perpendicular to the absorber
- Absorber should be thin and of uniform thickness
- The photon detector should have background count as low as possible.
The photon transmission measurements were done under a narrow beam counting geometry employing high resolution NaI(Tl) solid state detector. A NaI(Tl) scintillation detector (4.5cm diameter and 5.1cm thick) along a spectrometer assembly provided to allow maximum transmission of incident photons [5]. The experimental set-up used in the present work consists of mainly two collimators of diameter 0.2 cm which is well aligned by LASER beam so as to provide a scatter free collimated photon beam. The present experimental system was established from the photon spectrum that the energy of transmission photon did not charge appreciable due to scatter or fluorescent radiation from the collimators. A provision was made midway between the collimators to introduce absorbers which were in the form of thin foils. The entire system shown in Fig.3.2 was arranged vertically over the NaI(Tl) detector, ensuring that the central axis of the collimator coincided with the central axis of detector. The source is kept over the collimator so as to allow a narrow well collimated photon beam from the collimator incident normally on the thin absorber for the measurement of attenuation. Whole assembly is kept in the room to avoid any contribution of scattered photon from the walls. Care was taken to avoid any shift in the peak due to environmental changes. The Total photon interaction cross sections were measured using a narrow beam well collimated geometry setup (Murty and Devan, 1996) [6]. The gamma spectrum of 0.360MeV to 1.33MeV was measured before using collimator. The spectrums of high intensity keep constant and measure the linear attenuation coefficient (\(\mu\)), mass attenuation coefficient (\(\mu_m\)) and related parameter.
3.6 Synthesis of ferrite composite

Spinel ferrites are commercially important materials because of their excellent magnetic and electrical properties. Interesting physical and chemical properties of the magnetically diluted ferrites arises from the ability of these compounds to distribute the cations amongst the available tetrahedral A and octahedral B sites. Ferrites are able to fulfill a wide range of applications from microwave to radio frequencies, are a great importance from both fundamental and applied research point of view [7-8]. Recently, Ni$_{0.3}$Zn$_{0.5}$Cu$_{0.2}$Fe$_2$O$_4$ composite spinel ferrites were considered one of the most versatile magnetic materials for multilayer chip inductor (MLCI) applications and surface mount devices (SMDS) due to their high electrical resistivity, low sintering temperature and high permeability [9-10]. Researchers have been carried out about the synthesized Ni-Zn-Cu ferrites powder by various methods such as hydrothermal process [11], low fire process [12], sol-gel routes [13], and co-precipitation method [14] to overcome the problem in improving the performance of the ferrites.

3.6.1 Compounds used

The ferrite sample of composition Ni$_{0.3}$Zn$_{0.5}$Cu$_{0.2}$Fe$_2$O$_4$ were synthesized using standard ceramic method by mixing AR grade oxide of NiO, ZnO, CuO, Fe$_2$O$_3$ with proper proportion. All the oxides were 99.9% pure.

3.6.2 Method of preparation

The sample of Ni$_{0.3}$Zn$_{0.5}$Cu$_{0.2}$Fe$_2$O$_4$ ferrite system was prepared by solid-state reaction technique by using AR grade oxides of corresponding metals. The constituent oxides of the respective ferrite were, weighted
and mixed thoroughly. The mixture is well ground about 3 hours using agate mortar and pestle. The homogeneous mixture is then pressed into a circular pellet of 10 mm diameter and about 2 mm thickness using a hydraulic press. The samples in the pellet form were pre-sintered at 950°C for 12 hours in a programmable furnace. The samples are then slowly cooled to room temperature at the rate 2 °C per minute. The pre-sintered pellets were again crushed and reground to improve the homogeneity for about 2 hour. The dried mixture is compressed in circular pellet form. The polyvinyl alcohol (PVA) was used as a binder. These pellets were then sintered at 1100°C for 24 hours and finally slow cooled to room temperature at the rate of 2 °C per minute. The final obtained product in the form of pellet is hard, flat crack free and used for present investigation.

3.7 Characterization by XRD

It is very easiest method to understand the structure of prepared sample. X-ray diffraction analysis was carried out to assure the formation of the spinel structure of prepared sample. X-ray diffraction technique is a powerful tool to study the crystal structure of a material. Qualitatively and quantitatively it also determines the amorphous content of the sample. The technique helps in identifying the constituents of multiphase mixture. The X-ray diffraction pattern yields information of the position of Bragg’s peaks.

The XRD patterns also help to identify the structural phases present in the end product obtained by the method of preparation used. The XRD patterns of the samples were recorded on Philips X-ray
diffractometer (Model-3710). The XRD patterns obtained using Cu-Kα radiations (\(\lambda = 1.5405 \, \text{Å} \)) as a source at room temperature. The XRD patterns were recorded in the 2\(\theta\) range of 20\(^0\) to 80\(^0\) with scanning rate 1\(^0\) per minute. All the peaks in the recorded X-ray diffraction pattern are sharp intensive and in the prepared sample no extra peak is observed, it indicates that the prepared sample possess single phase cubic spinel structure. The Braggs peaks of the prepared sample is calculated by the XRD pattern using Bragg’s law,

\[n\lambda = 2d\sin\theta \quad \text{(3.2)}\]

Where,

- \(d\) is the inter planner spacing
- \(\theta\) is glancing angle (Bragg’s angle)
- \(\lambda\) is wavelength of incident radiation.
- \(n\) is an integer.
Table 3.1

Sources used to find the Gamma ray attenuation coefficient with their energies and peak channel number.

<table>
<thead>
<tr>
<th>Sr.No.</th>
<th>Source</th>
<th>Energy (MeV)</th>
<th>Peak channel No.</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>Ba$^{133}$</td>
<td>0.360</td>
<td>2.2</td>
</tr>
<tr>
<td>2</td>
<td>Na$^{22}$</td>
<td>0.511</td>
<td>3.2</td>
</tr>
<tr>
<td>3</td>
<td>Cs$^{137}$</td>
<td>0.662</td>
<td>4</td>
</tr>
<tr>
<td>4</td>
<td>Co$^{60}$</td>
<td>1.170</td>
<td>6.9</td>
</tr>
<tr>
<td>5</td>
<td>Na$^{22}$</td>
<td>1.280</td>
<td>7.4</td>
</tr>
<tr>
<td>6</td>
<td>Co$^{60}$</td>
<td>1.330</td>
<td>7.7</td>
</tr>
</tbody>
</table>
Fig. 3.1 Energy calibration curve at various photon energy versus peak channel number.
Fig. 3.2 Schematic diagram of Experimental apparatus used for measuring the mass attenuation coefficient
Fig. 3.3 Photograph of Experimental set-up of attenuation of gamma rays
REFERENCES


