

NEUTRON ACTIVATION ANALYSIS OF COMPOUNDS



By

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DEDICATION

To my mother Elfinesh Belayneh who has dedicated for the education of her children and could see only flower, but not lucky enough to see the fruits.

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Abstract

Neutron activation analysis(NAA) is used for quantitative determination and identification of elements from the chemical compound and rocks samples. Copper (Cu) and Iodine (I) and thier chemical compounds (CuSO_4 , KIO_4 and KI) were activated by using Am-Be neutron source and then the concentration of copper in CuSO_4 and iodine in KIO_4 and KI determined by comparative method. The sodium content of two rock samples obtained from the Department of Earth Sciences is identified by neutron activation of the rock samples. The proportion of the sodium content, as determined in this work, in the two rock samples agree with results obtained by inductively coupled plasma atomic spectrometry (ICP-AES) at CRPG, Nancy, France.

Chapter 1

GENERAL VIEW OF NEUTRON ACTIVATION ANALYSIS (NAA)

1.1 Introduction

Neutron activation analysis (NAA) is a method for the qualitative and quantitative determination of elements. The method is based upon the conversion of stable atomic nuclei into radioactive nuclei by irradiation with neutrons and the subsequent measurement of the radiation released by these radioactive nuclei. Amongst the several types of radiation that can be emitted, gamma radiation offers the best characteristics for the selective and simultaneous determination of elements i. e. neutron induced gamma ray emission is used for the identification and quantitative determination of constituent elements of a given sample.

The first analytical application of artificial radioactivity was carried out by G. Hevery and H. Levi from 1936 to 1938 and a systematic development of activation analysis only began in the years around 1950 (Erditmann and Petri, 1983). NAA is now commonly used in industry to identify certain metals in a sample and in forensic application to identify a sample uniquely by the use of trace constituent elements.

As compared to other analytical methods, NAA is a highly sensitive analytical technique and requires only a minimum of sample handling and treatment before irradiation, which gives it the following advantages:

NAA is non-destructive NAA enables the direct analysis of compact pieces like coins, precious stones, seed grains or other small organs from plants and animals with out destruction and in many cases samples can be used for further investigations.

Minimized loss and Contamination of trace elements The risk of contamination and trace element loss, which are the most serious sources of error in trace element

analysis, are minimal in the case of NAA. With this method compact lumps of rocks, ceramics or other hard materials with freshly broken, untouched surfaces can be directly irradiated and measured.

Ease of calibration and accuracy This is due to the fact that the neutrons used for activation as well as the gamma rays used for measurement thoroughly penetrate the sample materials. So that the relation between element concentration and measured activity is nearly matrix-independent. That means, the analytical sample and reference sample need not consist of the same matrix element as required for many other methods and activation analysis can be applied directly to a great number of different sample materials.

NAA requires the use of large irradiation machines, fission type reactors or particle accelerator at least if trace element determinations have to be carried out. This disadvantage prevents many analytical laboratories from applying this method. However, a good many investigation can be done with long lived radionuclide in laboratories far from these facilities. A more stringent handicap is the fact that these laboratories must be especially equipped for handling radioactive material according to prescribed safety regulations. Another drawback is the long lived time required for some analysis where long lived radionuclide are involved. For measuring the spectrum of nuclear radiation from activation products is carried out using high resolution gamma ray spectrometry. Especially, gamma ray detectors made from highly pure germanium (HPGe) are most preferable.

In this study, NAA is used to identify constituent elements of known compounds and rock sample of known composition. The compounds are from the physics and chemistry departments of the AAU where as the rock samples were obtained from the Earth science department of AAU.

Major elements in the rock samples were analyzed and identified by inductively coupled plasma atomic emission spectrometry (ICP-AES) at CRPG, Nancy, France and the results of the measurement were published in (Ayalew and Yirgu, 2003)

This first chapter deals with the general view of NAA. This encompasses, historical, theoretical and technical developments; current status and applications of NAA.

In the second chapter the interaction mechanisms of gamma rays and neutrons with matter are briefly revised.

The working principle of ionizing radiation detectors especially scintillation and Ge detectors are described in third chapter. In chapter 4 the experimental equipment and methods used in the experiment are discussed. Chapter five presents the data, analysis and conclusion drawn in this work.

1.2 NAA: Fundamentals

When a neutron interacts with the target nucleus via a non-elastic collision, a compound nucleus forms in an excited state. The compound nucleus will almost instantaneously de-excite in to a more stable configuration through emission of one or more characteristic prompt gamma ray. In many case, these new configuration yields a radioactive nucleus which also de-excites (or decays) by emission of one or more characteristic delayed gamma rays, but at a much slower rate according to the unique half-life of the radioactive species, half-life can range from fraction of a second to several years.

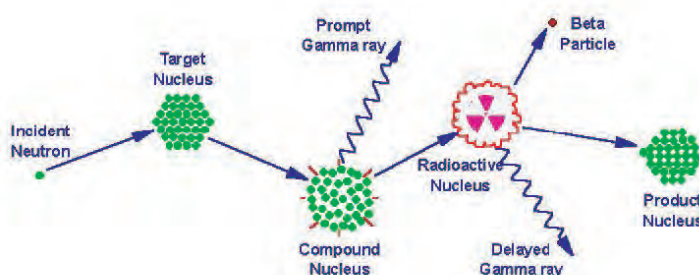


Figure 1.1: Neutron capture by a target nucleus followed by the emission of gamma rays

The radio activity induced can, uniquely, be characterized by half lives of the radio nuclides produced and by the radiation emitted. The decay rate and radiation energies are never duplicate in any two radioisotopes produced by neutron irradiation. Among more than 5000 known isotope about 95% are radioactive (Baxter, 1993). Hence an individual nuclear “fingerprint” can be measured and quantified (Tequash, 1995).

The rate of formation of a particular activation product, R_F in a given sample is proportional to the incident neutron flux density, to concentration of the target nuclide in the irradiated sample and to the cross-section for the nuclear reaction. Thus:

$$R_F = \phi \sigma n = \frac{\phi}{A} \sigma m f N_o \quad (1.1)$$

where

ϕ = the neutron flux

f = the fractional isotope abundance of target nuclide

σ = the reaction cross-section

A = the atomic weight of the trace element

n = the # of accessible target atoms

m = the mass of the trace element in the specimen

N_o = Avogadro number.

The decay rate, D , expressed in atomic disintegration per second of the product radionuclide in the specimen is given by:

$$D = n\lambda \quad (1.2)$$

where n is the number of atoms of the nuclide in specimen and λ is the decay constant of the nuclide. Therefore, the rate of change of the product during irradiation is:

$$\frac{dn}{dt} = R_f - D = \frac{\phi}{A} \sigma m f N_o - n\lambda \quad (1.3)$$

The disintegration rate in the specimen after an arbitrary irradiation time T is:

$$D(t) = \frac{\phi}{A} \sigma m f N_o (1 - \exp(-\lambda T)) \quad (1.4)$$

For sufficiently long irradiation time $\exp(-\lambda T)$ approaches zero, so that equation (1.4) becomes:

$$D(t_\infty) = \frac{\phi}{A} \sigma m f N_o \quad (1.5)$$

Eq (1.4) can also be written in the following form for calculating the sensitivity for detection an element under specific set irradiation and detection condition:

$$m = \frac{AD_t}{\phi \sigma f N_o (1 - \exp(-\lambda T))} \quad (1.6)$$

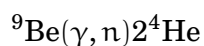
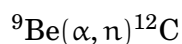
1.3 Neutron Sources

Although there are several types of neutron source (reactor, accelerator and radioisotope neutron emitters). The most convenient choice for university's laboratory work is isotopic neutron sources. Because, they have the unique advantages of being small, portable, reliable and relatively cheap. There are two types of radioisotope neutron sources:

Spontaneous fission: is a decay mode of some transuranic isotopes during which two to four neutrons are released during this process. Cf a common example of such sources.

Two-component sources: based on (α, n) and (γ, n) reactions. They are composed of a long-lived radioactive isotope supplying alpha particles or gamma photons and a target material of low atomic number.

The neutrons are obtained by nuclear reaction as:



The main disadvantage of neutron sources of the latter type is the fact that very large gamma-ray activities must be used in order to produce neutron sources of attractive intensity, therefore, the neutrons appear in a much more intense gamma-ray background.

Radioisotope sources

Due to energetic alpha particles are available from the direct decay of a number of convenient radionuclides, it is possible to fabricate a small self-contained neutron source by mixing an alpha-emitting isotope with a suitable target material. Several different target material can lead to (alpha,n) reactions for alpha particle energies that are readily available in radioactive decay. The maximum neutron yield is obtained when beryllium is chosen as the target. Neutrons are produced through the reaction



which has a Q-value of +5.71MeV. All the alpha emitters of practical interest are actinide elements, and investigation shown have that a stable alloy can be formed between the actinids and beryllium of the form $M\text{Be}_{13}$, where M represents the actinide metal. Most of the sources listed in the table below therefore, are metallurgically prepared in the form of this alloy, and each alpha particle has an opportunity to interact with beryllium nuclei without any intermediate energy loss.

Table 1.1: Characteristics of Be(α ,n) neutron source (Knoll, 2000)

source	half-life	E_{α} (MeV)	neutron yield per 10^6 α -particles ¹
²³⁹ Pu/Be	24000y	5. 14	57(exp)
²¹⁰ Po/Be	138d	5. 30	69(exp.)
²³⁸ Pu/Be	87. 4y	5. 48	79(cal.)
²⁴¹ Am/Be	433y	5. 48	70(exp.)
²⁴⁴ Cm/Be	18y	5. 79	100(cal)
²⁴² Cm/Be	162d	6. 10	106(exp.)
²²⁶ Ra/Be	1602y	multiple	502(cal.)
²²⁷ Ac/Be	21. 6y	multiple	702(cal.)

The radioisotopes in Table 1.1 except Ra and Ac involve simpler alpha decays and the gamma-ray background is much lower. The choice between these alternatives is made primarily on the basis of availability, cost and half-life. Due to this, Am(half-life of 433 years) and Pu(half-life of 87.4 years) are widely used if high neutron yields are needed.

Determination of elemental composition in a sample

For many evaluations of γ -ray spectra the radio nuclide report is the final step. With activation analysis, however, the analytical results must be presented as a list of the elements found and their amount in grams or their concentration in grams per gram of sample. Although, there are different kinds methods of evaluation the absolute method and the relative method are most common.

Absolute method

The absolute method requires suitable calibration, i.e. the spectrometer must be calibrated with respect to the energy scale and peak counting efficiencies.

If an amount m of an element is irradiated with a thermal neutron flux ϕ_{th} for an irradiation period of t_{irr} then at the end of irradiation the activity or decay rate A_o of a radio nuclide built up from (Erditmann and Petri, 1983):

$$A_o = N_o \frac{m\alpha}{M} \phi_{th} \sigma_{th} [1 - \exp(-\lambda t_i)] \quad (1.8)$$

Where, N_o is Avogadro's number, M is the atomic weight of the element, α is the abundance of the isotope yielding the radio nuclide to be measured, σ_{th} is the thermal cross section λ is the decay constant of the radio nuclide.

The activity after a waiting period t_d , also known as delay time, is measured by the gamma-ray Spectrometer and from the counts (P) accumulated in the photo peak during the counting period t_m . A_o can be calculated as:

$$A_o = \frac{P\lambda \exp(\lambda t_d)}{h\eta [1 - \exp(-\lambda t_m)]} \quad (1.9)$$

Where h is the gamma-ray abundance and η is the photo peak counting efficiency.

In the following equations of this section the time dependent terms will be abbreviated according to the following:

$$1 - \exp(-\lambda t_i) = B(\text{build - up term}) \quad (1.10)$$

$$\frac{\lambda \exp(\lambda t_d)}{1 - \exp(-\lambda t_m)} = D(\text{decay and measuring term}) \quad (1.11)$$

If the half-life $t_{1/2} = \frac{\ln 2}{\lambda}$ is longer than $\simeq 3t_m$, the decay and measuring term is simplified to:

$$\frac{\exp(\lambda t_d)}{t_m} = D \quad (1.12)$$

If all entities are accurately known the expected, activity from Equation(1.6) and the measured activity from equation (1.8) are equal and the amount of element can be calculated:

$$m = \frac{\text{PMD}}{N_o a h \eta \phi_{th} \sigma_{th} B} \quad (1.13)$$

The calculation of analytical result from equation (1.13) is called the absolute method. A 20-50% uncertainty was to be expected if analysis were carried out by this method using equation (1.12) This was due to the fact that tabulated data for gamma-ray abundance and reaction cross-section from different sources were used (Erditmann and Petri, 1983).

The Relative Method

The application often absolute method suffered for a long time from uncertainties in the nuclear data. Therefore, the relative method was preferred since it yielded the most reliable results. And it seems till to be the most accurate method if accurate reference samples are available. With the relative method flux ratio measurements are not required and no cross-section value are needed. It avoids also the efficiency calibration.

To determine an element i in a sample one prepares reference sample with an accurately known content of elements i irradiates both samples simultaneously and measures their activities under the same conditions with the gamma ray spectrometer. Then from the photo peaks pertaining to element i . One can immediately calculate its amount in the sample being analyzed (Erditmann and Petri, 1983).

$$m_{i,s} = m_{i,r} \frac{A_{o,s}}{A_{o,r}} \quad (1.14)$$

Where $m_{i,s}$ and $m_{i,r}$ are the amount of element in the sample and in the reference sample respectively. The A_o 's are the decay rates at some reference point of time, usually the end of the irradiation period is chosen. From the peak areas P_s and P_r that is the number of counts registered in the photo peak. Pertaining to element i during the measuring periods $t_{m,s}$ and $t_{m,r}$ one obtains:

$$\frac{A_{o,s}}{A_{o,r}} = \frac{P_s D_s B_r}{P_r D_r B_s} \quad (1.15)$$

Where B and D correspond to Equation 1. 8 and 1. 9 or 1. 10. the relative method is the simplest method if only a few elements have to be determined and it is the most precise method if precise reference samples are used.

1.4 NAA: Techniques

Techniques in NAA fall in to two categories, on the basis of the time of measurement.

1. Prompt gamma-ray Neutron Activation Analysis(PGNAA), where measurement take place during irradiation.
2. Delayed Gamma-ray Neutron Activation Analysis(DGNAA), where the measurement follow radioactive decay. This operation mode is more common thus,when one mention NAA it is generally assumed that measurement of the delayed gamma rays is intended.

DGNAA is useful for the vast majority of elements that produce radioactive nuclides. The technique is flexible with respect to time such that the sensitivity for a long-lived radionuclide that suffers from an interference by a short-lived radionuclide can be improved by waiting for a short-lived radionuclide to decay. With the use of automated sample handling, γ -ray measurement with solid-state detectors and computerized data processing it is generally possible to simultaneously measure more than thirty elements in most sample types without chemical processing. The application of purely instrumental procedures is commonly called instrumental Neutron Activation Analysis (INAA) and is one of NAA'S most important advantages over other analytical technique. If chemical separations are done to sample after irradiation to remove interference or to concentrate the radioisotope of interest,the technique is called Radiochemical Neutron Activation Analysis (RNAA).

1.5 Sampling for NAA

The aim of sampling is to take a representative sample of an object to be characterize. It must be representative at least with respect to the properties that are to be measured. In analytical chemistry that means that the chemical composition of the sample elements, compounds, phases or what ever was to be determined must be the same as in the whole material to be characterized. Obviously it is very difficult to obtain a representative sample small enough to be analyzed from a large in homogeneous amount of material.

From samples being prepared and homogenized by grinding ,sieving and mixing the amount to be used for a representative analysis depends on the grain size of the sample and on the concentration of the elements to be determined.

1.5.1 Sample size and shape

The amount of sample to used depends on many factors including total amount of sample material available, its homogeneity the concentration of elements to be measured and the neutron absorption and activation cross–Section of the matrix materials. Samples between

100 and 1000 mg are convenient to handle, but if the irradiation contains is large enough up to 100g of material of even more may be irradiated. On the other hand with rare and expensive materials under an advantageous circumstances even a few micrograms will be sufficient for farce element determination.

The shape of samples is usually not critical either. Where as the other instrumental methods require, for example, finely ground powders or will prepared, smooth surface or specially machined pieces fitting the sample holders, instrumental neutron activation analysis can feat samples of very different shapes as long as their dimensions do not exceed since large pieces are not uniformly activated due to the neutron flux gradient in the source or to neutron self shielding.

1.5.2 Sample preparation

Once a representative sample has been identified from the material to be characterized it is usually necessary to take sub samples and to prepare them for irradiation in the neuron source. Sample preparation steps are include drying, ashing or pre-concentration. The pre-concentration of one or few elements may become necessary if the elements to be measured are very short-lived so that there is not enough time for a post irradiation preparation.

All these during ashing and pre-concentration procedures bear a higher risk of contamination and losses of constituents. The risk of contamination may come:

- from the tools and containers used for treatment and storage of the sample by product of abrasion often metal dust become mixed with the sample.
- From air dust laboratory dust or dust from material stored or handled in the vicinity.
- From the experimenter her or himself (Sweat, Skin particles, debris from clothing, cosmetic substance).
- From reagents and solvents used .
- From reaction of the sample surface with oxygen and nitrogen.

On the other hand constituents may be lost be volatilization, or with dilute solution, by absorption on the container wall.

There are many laboratory technique to control or avoid the risk of contamination. Hard corrosion resistance tools should used for sampling, cutting, crushing and grinding. But with hard sample material like rocks, soil or ceramics, abrasion cannot be avoided. With sample type where the abrasion effect is especially high it may be better to avoid crushing and grinding. If the sample material is sufficiently homogenous it may be dashed in to pieces small enough to it in the irradiation container by using a hard tool. Then pieces with fresh and untouched surface may be selected. The tools are made from materials not

to be measured in the sample; for example a titanium knife may be used if titanium is not be determined and does not interfere with other elements.

The vessels used for storage, dissolution and treatment of the samples must be made from very pure materials, such as polyethylene and synthetic silica.

1.6 Applications

The applications for NAA are practically limitless for solid, liquid, or gaseous samples. Recent applications include:

- Environmental studies to characterize pollutants and determine their source and methods of reduction.
- Semiconductor materials analysis to measure ultra trace-element impurities and to determine methods for reducing or eliminating impurities from final products.
- Pharmaceutical materials analysis to measure ultra trace-element impurities and to determine methods for reducing or eliminating impurities from final products.
- Forensic studies as a non-destructive method to analyze evidence as an aid in the investigation and prosecution of criminal cases.
- Archaeological studies to fingerprint artifacts and determine the place of origin as a way to understand the activities of humans in the past. Nutritional epidemiological studies to investigate the contribution of diet, occupation, and lifestyle on chronic diseases.

Chapter 2

Radiation Interaction

The radiation of primary concern in this context originate in atomic or nuclear processes. They are conveniently categorized in to four general type as follows:

Charged particle radiation: Fast electrons and heavy charged particles. Fast electrons include beta particles(positive or negative)emitted in nuclear decay, as well as energetic electrons produced by any other process.

Heavy charged particles denote a category that encompasses all energetic ions with mass of one atomic mass unit or greater, such as alpha particle, protons, fission products, or the products of many nuclear reactions.

Uncharged radiation: Electromagnetic radiation and Neutrons. The electromagnetic radiation of interest include X-rays emitted in the rearrangement of electrons shells of atoms, and gamma rays that originate from the transitions within the nucleus itself.

Neutrons: neutrons generated in various nuclear processes constitute the final major category which is often further divided in to slow neutrons and fast neutrons subcategories as mentioned in chapter one.

Because of the operation of any radiation detector basically depends on the manner in which the radiation to be detected interacts with the material of the detector itself, an understanding of the response of a specific type of detector must therefore be based on a familiarity with the fundamental mechanisms by which radiation interact and lose their energy in matter. Therefore, for interest of this paper it is better to describe in detail about interaction of gamma rays and neutron.

2.1 Interaction of gamma ray

There are a number of processes which can cause gamma rays to be scattered or absorbed. A catalogue of the possible processes by which the electromagnetic field of the gamma ray may interact with matter has been put in the following systematic form by Fano (Evans, 1955).

Table 2.1: Interaction of Photons with matter

kinds of interaction	Effects of interaction
1) Interaction with atomic electrons	a) Complete absorbtion
2) Interaction with nucleons	b) Elastic scattering
3) Interaction with the electric field	c) Interaction with the electric field
4) Interaction with the meson field	

There are 12 ways of combining column 1 and 2, thus in theory there are 12 different processes by which gamma-ray can be absorbed or scattered. Many of the process are quite infrequent and some have not yet been observed. Most frequently the major effects are explained in terms of just three of the above 12 processes. These are the photoelectric effect (1a), the Compton effect (1c) and pair production (3a) (Evans, 1955).

2.1.1 Photoelectric effect

In the photoelectric effect process, a photon undergoes an interaction with an absorber atom in which the photon completely disappears. In its place, an energetic photo-electron is ejected by the atom from one of its bound shells. The interaction is with the atom as a whole and can not take place with free electrons. For gamma-rays of sufficient energy, the most probable origin of photoelectron is the most tightly bound or K shell of the atom. The photoelectron appears with an energy given by

$$E_e = h\nu - E_B \quad (2.1)$$

E_B is the binding energy of the electron and $h\nu$ is the energy of the incident photon.

For gamma-ray energies of more than a few hundred KeV, the photoelectron carries off the majority of the original photon energy (Knoll, 2000).

In addition to the photoelectron, the interaction also creates an ionized absorber atom with a vacancy in one of its bound shells. This vacancy is quickly filled through capture of free electrons from the medium and/or rearrangement of electrons from other shells of the atom. Therefore, one or more characteristic X-ray photons may also be generated.

Although, in most cases these X-rays are reabsorbed close to the original site through photoelectric absorption involving less tightly bound shells, their migration and possible

escape from radiation detectors can influence their response.

The photoelectric process is the predominant mode of interaction for gamma-rays(or X-rays)of relatively low energy. The process is also enhanced for absorber material of high atomic number Z .

No single analytic expression is valid for the probability of photoelectric absorption per atom, over all range of E_γ and Z , but a rough approximation is: (Knoll, 2000)

$$\tau \cong \text{constant} \times \frac{Z^n}{E_\gamma^{3.5}} \quad (2.2)$$

Where the n varies between 4 and 5 over the gamma-ray energy region of interest.

This severe dependence of the photoelectric absorption probability on the atomic number of the absorber is primary reason for the preponderance of high- Z constituents because of the high absorption for this radiation.

2.1.2 Compton Scattering

The interaction process of Compton scattering takes place between the incident gamma-ray photon and an electron in the absorbing material. It is most often the predominant interaction mechanism for gamma ray energies typical of radioisotope sources (Knoll, 2000).

In Compton scattering, as shown below the incoming gamma-ray photon is deflected through an angle θ with respect to its original direction. The photons transfers a portion of its energy to the electron(assumed to be initially at rest), which is then known as a recoil electrons. Because all angle of scattering are possible, the energy transferred to the electron can vary from zero to a large fraction of the gamma-ray energy.

The energy transferred to the electron vary from zero (when $\theta = 0$) to maximum energy E_m (when $\theta = \pi$).

For small scattering angle θ , very little energy is transferred. Some of the original energy is always retained by incident photon, even in the extreme of $\theta = \pi$.

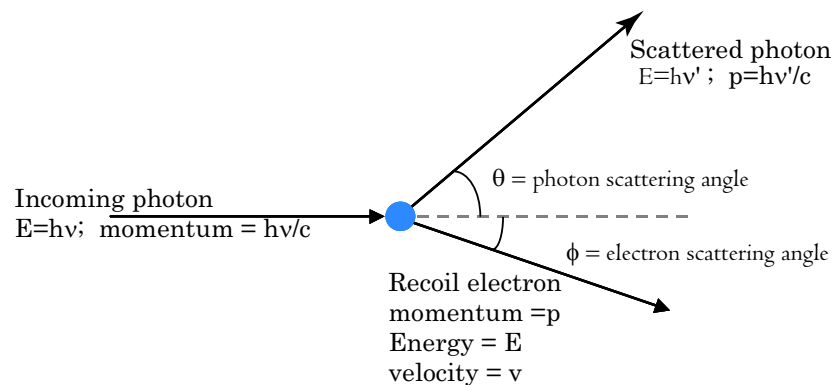


Figure 2.1: Events in the Compton scattering process.

2.1.3 Pair-Production

The third mechanism by which electromagnetic radiation can be absorbed is the production of electron-positron pair. This effect is most prominent above incident photon energy of 1.02 MeV. In this type of interaction, the photon is completely absorbed and in its place appears a positron-electron pair whose total energy is just equal to $h\nu$.

$$h\nu = (T_- + m_0C^2) + (T_+ + m_0C^2) \quad (2.3)$$

where T_- and T_+ are the kinetic energies of the electron and positron respectively and $m_0C^2 = 0.511 \text{ MeV}$ is the electronic rest mass energy. The process occurs only in the field

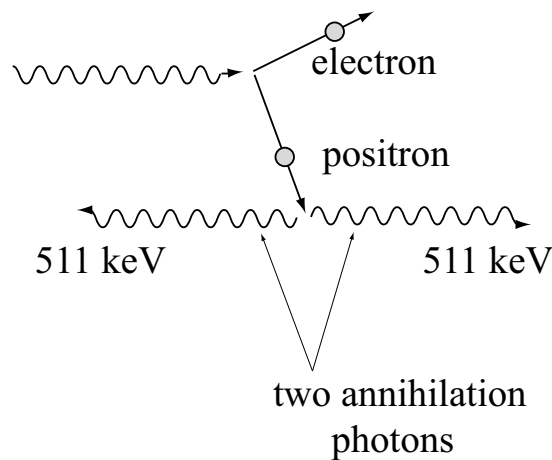


Figure 2.2: Pair production

of charged particles, mainly in the nuclear field but also to some extent in the field of an electron because somebody must be given recoil energy and momentum in order that energy and momentum can be conserved in the system. The cross-section is zero for photon energies less than 1.02 MeV, for greater energies, it increases at first slowly then more rapidly. It is proportional to Z^2 . For a given photon energy, pair formation increases quite rapidly with atomic number and probability for pair production is given by the following expression:

$$\text{Probability}_{(pp)} = k (\log(h\nu)) (Z)^2 \quad (2.4)$$

The process of pair-production is closely related to the reverse process, that of electron-positron annihilation. A positron after being formed is slowed down by collisions with atoms, until it is practically at rest. It then interacts with an electron which is also practically at rest. The two particles disappear and two photons appear moving in opposite

directions, each with an energy of 0.511 MeV equal to the rest energy of an electron.

$$e^+ + e^- \rightarrow 2h\nu \quad (2.5)$$

The two gamma-rays in equ (2.5) are called annihilation radiation (Lapp, 1972).

The photons which appear on annihilation of an electron-positron pair are called annihilation photons and the absorption of gamma-rays by the pair-production is always complicated by the appearance of two low energy secondary radiation.

2.2 Neutron Interaction

The behavior of neutrons in matter is quite different from that of gamma rays or charged particles. Since the neutrons are uncharged, no coulomb forces come in to play with either the orbital electrons or the nuclei.

For neutrons to interact with matter, i.e. for the nuclear force to act, they must either enter the nucleus or come sufficiently close to it. The type of the interaction taking place between a neutron and the nucleus differs depending upon the kinetic energy of the incident neutron.

2.2.1 Energy Classification

For the purpose of study of neutron interactions; neutrons are classified as below: (Glascock, 2005)

- Thermal neutrons Energy below 0.5 eV
- Intermediate neutrons 0.5 eV – 100 keV
- Fast neutrons 100 keV – 20 MeV
- High energy neutrons above 20 MeV.

Study of the neutron interaction with matter requires the knowledge of neutron energy spectrum. For many applications the spectrum is poorly known. All neutrons are fast by birth and lose energy by colliding elastically with atoms in their environment and then after being slowed down to thermal energies they are captured by the nuclei of the absorbing medium.

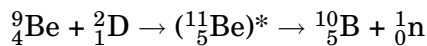
2.2.2 Neutron Sources

The most prolific source of neutrons is the nuclear reactor. The splitting of a uranium or a plutonium nucleus in a nuclear reactor is accompanied by the emission of several neutrons.

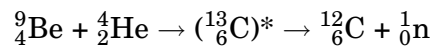
These fission neutrons have a wide range of energies, peak at ≈ 0.7 MeV and have a mean value of ≈ 2 MeV.

Californium 252, an alpha emitter, is another source of neutron. It undergoes spontaneous nuclear fission at an average rate of 10 fissions for every 313 alpha transmissions. The neutron emission rate has been found to be 2.31×10^6 neutrons per second per microgram of ^{252}Cf . The emitted neutrons span a wide range of energies. The most probable energy is about 1 MeV, while the average value of the energy distribution is about 2.3 MeV.

Copious neutron beams may be produced in accelerators by many different reactions. For example, bombardment of beryllium by high-energy deuterons in a cyclotron produces neutrons according to the reaction:



Another commonly used neutron source depends on the bombardment of beryllium with alpha particles. The reaction, in this case, is



Radium, polonium, and plutonium are used as the source of the alpha particles. Photoneutrons are the other important source of neutrons. In this process gamma radiations from ^{24}Na , ^{226}Ra , ^{124}Sb , ^{72}Ga and ^{140}La bombarding ${}^9_4\text{Be}$ and ${}^2_1\text{H}$ give neutrons (having an energy distribution).

2.2.3 Interaction Mechanisms

There are a number of processes by which a neutron can interact with matter.

1. **Elastic scattering (n,n)** In elastic collision both the momentum and kinetic energy of the system of neutron and interacting nucleus are conserved. The process may be regarded as essentially a billiard ball type of collision. In each collision with a stationary nucleus, the neutron transfers part of its kinetic energy to the nucleus depending on the angle through which the neutron is scattered.
2. **Inelastic scattering** In the range of energy above 0.5 MeV inelastic scattering begins to occur [(n, n), (n, 2n), (n, γ) type of reactions]. In this case a part of kinetic energy of the incident neutron is given off in the form of one or more gammas. This process always takes place through the formation of compound nucleus. This type of interaction is not possible unless the neutron energy exceeds a certain threshold
3. **Nonelastic scattering (Nuclear Reactions Involving Emission of Charged Particles)** Nonelastic reactions occur at high neutron energies [(n, α), (n, p) type]. These are the

reactions with energy thresholds in which the neutron causes the emission of charged particles (protons or other heavier particles) from the target nucleus. An example of particular importance in biological tissue is the (n, p) reaction of ^{14}N with slow neutrons: $^{14}_7\text{N} + {}^1_0\text{n} \rightarrow {}^{16}_6\text{C} + {}^1_1\text{H}$

This reaction produces a proton of 0.58 MeV energy.

4. **Radiative Capture (n, γ)** Nuclear process in which a neutron is captured by the target nucleus and the excess energy emitted as radiation, is called radiative capture process. Conditions for such reactions are especially favorable during slow neutron (with energy $< 1\text{ eV}$) interaction with medium. Cross section for these processes usually decreases with the inverse of the neutron velocity. It is a very common process, for it occurs with a wide variety of nuclides from low to high mass numbers. This process is extensively used for the production of isotopes by exposing stable nuclides to slow neutrons in a nuclear reactor.
5. **Spallation reaction:** In this process the target nucleus is fragmented with the emission of several particles often including neutrons. The process becomes significant only at neutron energies of about 100 MeV or greater.
6. **Nuclear fission (n,f):** In certain reactions involving heavy atomic nuclei, the capture of neutron results in the formation of an excited state of a compound nucleus so unstable that it splits up into two smaller nuclei. This process is of fundamental importance for the operation of nuclear reactors.

2.2.4 Cross-section

Atomic nuclei occupy only a very small space within matter and neutrons can penetrate thousands of atomic layers before they hit an atomic nucleus. The nuclei seen from the neutrons can be regarded as small discs whose areas are the cross sections. The unit is cm^2 . The values of total cross sections are usually in the order of 10^{-24} cm^2 which is approximately the size of an atomic nucleus. Therefore, 10^{-24} cm^2 (one barn) is often used as the unit for cross section values. Some nuclei, however, have cross sections much greater than their nucleus. This can be explained by long range forces which influence the neutron even if it passes the nucleus at a large distance. Some nuclei of rare earth elements have cross sections, of more than 1000 b for slow neutrons.

When a neutron has hit a nucleus it may be scattered or absorbed. The total cross section can be divided into two parts according to the probabilities of the two reactions.

$$\sigma_t = \sigma_s + \sigma_{abs} \quad (2.6)$$

The absorption cross section can be subdivided into the reaction cross sections according to the fate of the compound nucleus.

$$\sigma_{\text{abs}} = \sigma_{(n,\gamma)} + \sigma_{(n,p)} + \sigma_{(n,2n)} + \sigma_{(n,n)} \quad (2.7)$$

When metastable states exist, the (n, r) cross section can be further subdivided into those leading to different states:

$$\sigma_{(n,\gamma)} = \sigma_{(n,\gamma)g} + \sigma_{(n,\gamma)m1} + \sigma_{(n,\gamma)m2} + \sigma_{(n,\gamma)m3}\dots\text{etc.} \quad (2.8)$$

Where the index g indicates the ground state and $m1$, $m2$, $m3$, etc the metastable states. From the above equations it is obvious that reaction cross section can not be correlated with the area of the atomic nuclei. They are reaction constants with the dimension of an area (Erditmann and Petri, 1983).

$$R = \frac{-dn}{dt} = \phi\sigma N \quad (2.9)$$

Where R is the reaction rate N is the number of the target atoms and ϕ is the neutron flux.

The size of the cross section depends on the velocities or energies of the neutrons. One can imagine that a slowly moving neutron stays within the sphere of influence of an atomic nucleus for a long time and its probability of reacting is greater than that of a fast neutron. Indeed it is found that at low energies the reaction cross sections for most nuclides are inversely proportional to the velocity of the neutron.

The thermal (n, γ) cross-section varies by large factors from one isotope to another and thus from element to element. Also, γ -ray emission probabilities and half-lives can vary by large factors between radionuclides. Therefore, the sensitivity of NAA varies strongly with the element involved. Some elements, in particular light ones, can not be detected by thermal NAA (i.e. H, C, N, O, Si, P, S and Ca) (Debertin and Helmer, 1988).

Chapter 3

Radiation Detector

A detector system can be considered to consist of two parts, a detector and a measuring apparatus. The interaction of the radiation with the system takes place in the detector. The measuring apparatus takes the out put of the detector and performs the functions required to accomplish the measurements.

Detectors can be characterized by the nature of the interaction of the radiation with the detector. Several types operate by virtue of ionization which is produced in them by the passage of radiations. For uncharged radiations such as neutrons and gamma-rays, the charged particles that are required for ionization are obtained by secondary processes.

The detection of radioactivity is made possible through the interaction of radiation with matter. In interacting with matter, the various radiations directly or indirectly produce excitation or ionization of atoms of matter. The mechanism of interaction of two radiations with matter were discussed, these are and gamma-ray radiation.

Neutrons are neutral particles and do not interact with matter electro-magnetically. In order to detect fast neutrons, one places a hydrogenous material in their path and detect the recoil protons which were knocked out through the elastic collision between the fast neutrons and the hydrogen nuclei. Slow neutron, being neutral and traversing matter slowly, have very large probability of capture by nuclei. The product resulting from their capture are either heavy ionizing particle (such as α - particles, protons, or fission fragments) or γ radiation. The detection of these products constitutes the essential means of detecting slow neutrons.

The gamma ray (γ) is an electromagnetic radiation, which does not produce ionization in matter directly. However, γ ray interacts with matter by producing either photoelectron in photoelectric effect, a recoil electron in the Compton effect, or an electron-positron pair in the process of pair production. The photoelectron, Compton-recoil electron, or electron-positron pair thus produced are fast-moving charged particles and therefore can be used in the detection of gamma ray. Now we will see the different kind of detector.

3.1 Scintillation Detector

There are several substances in certain organic and inorganic materials, which emit light flashes or scintillation when charged particles ray or gamma -ray pass through them.

These substances are called *scintillator*. In a scintillation detector these light flashes are allowed to fall on the photo cathode of a photo multiplier tube and a pulse is extracted out to Signal of the passage of nuclear radiation. The height of this output pulse can be made proportional to the energy dissipated by the ionizing radiation in the scintillator. Thus a scintillation detector can be used not only for counting but for energy analysis also. Because of its versatility, there has been considerable development of scintillation detectors. Before we describe the operations of a scintillation detector. We will see how scintillation will be produced

3.1.1 Production Of Scintillation

When a fast ionizing particle passes through a crystal, it can excite electrons within an atom or a molecule in to higher states. The excitation energy can be given off in different ways. Some of the more important processes are:-

1. The electrons can fall back to the lower states with emission of light in characteristic lines or bands- fluorescence.
2. The system (atom or molecules) may go over into a metastable state from which delayed light emission is transfers the excitation energy to some trapping counter(crystal imperfection, impurities or "activators") from which it can be released only after thermal activation. The delayed "persistent" light emission is called *photophorescence*. In the case of scintillation (light flash) produced by a simple particle can be seen only if the delay of the phosphorescence is sufficiently short otherwise the total amount of light will be distributed over a long time and the light intensity will be too low to be detectable.

3.1.2 Operation Of Scintillation Detector

The other thing is the operation of scintillation detector. the main operation of scintillation detector is as follows. We shall describe the operation of a scintillation detector step by step.

1. *The absorption process:*

A charged particle falling on a scintillation can dissipate all its energy in its if the

dimensions of the scintillator are large compared to its range. if a gamma ray (x-ray) is incident on a scintillator it may interact with matter in three ways namely Photoelectric, Compton and pair production interactions.

In each of this effect, electrons are produced and by successive interactions the gamma rays can transfer all or part of its energy in to the kinetic energy of such secondary electrons. These secondary electrons will give up their kinetic energy in ionization or excitation in the scintillation material.

2. *The scintillation process:*

The scintillator absorbs energy when it undergoes ionization and excitation by the electrons. This absorbed energy appears either as heat energy or as luminescence photons. In the latter process, the excited states of the scintillator material de-excite to lower states by light emission within 10^{-8} seconds or less. This emitted light is known as scintillation. The properties of some commonly used scintillator is NaI(Tl). We will see deeply about NaI (Tl) crystal.

3. *Conversion of light into electrical pulse:*

The light emitted by the scintillation is allowed to fall on the photocathode of a photomultiplier tube, which is optically coupled to the scintillator. The scintillation is enclosed in an envelope having reflecting walls and the scintillator is transparent to its own radiation. as a result depending on the geometry and the optical properties of the system certain fraction of light produced in the scintillator falls on the photocathode of the photomultiplier tube producing photoelectrons. The structure of a photomultiplier tube is such that there are several dynodes and these are maintained at successively higher electrical potential (about 100 volt per dynode stage) further the dynode surfaces have a property of emitting more(say 3-5) secondary electrons when one incident electron strikes them. Thus a photoelectron emitted by a photocathode is accelerated by the electric field to the first dynode where it produces a bunch of secondary electrons. These electrons are now accelerated towards the next dynode. where they intern, produce more electrons and this process is repeated at each dynode.

In a photomultiplier tube there are usually 10 or more dynodes stages and one can usually achieve again up to 10^7 - 10^8 by the time electrons reach the last stage which is called the anode. In this way the initial ionizing radiation gives rise to a burst of electrons at the anode where an electrical pulse is taken out for further analysis.

Many photomultiplier tubes have been developed commercially and today one has a choice of several versatile photomultiplier tubes. The photocathode in these tubes is usually a semitransparent coating of some photoemission material. (e. g antimony-caesium)

on the inner surface of the flat top of the tube envelope . The quality of a photomultiplier tube is mostly determined by two properties (i) the transit time or the time taken by the electrons to reach the anode through the dynode. structure. this property is important for the timing characteristic of the photomultiplier tube (ii) the stability of the electron multiplication factor. This property determines the energy resolution characteristics of the photomultiplier tube.

3.1.3 scintillation gamma rays spectrometer

One of the most efficient method of counting gamma rays and measuring their energies is their detection by a scintillation gamma ray spectrometer. This spectrometer employs a scintillation detector which is usually a thallium activated sodium iodide [NaI (Ti)] crystal as a scintillator. The NaI (Ti) crystal is sealed in an aluminium can. A glass window is provided at one end so that light produced by the scintillator can pass on to the photocathode. The NaI (Ti) crystals are usually in the form of right circular cylinders, their dimension being determined by the needs of the particular measurement. A gamma ray spectrometer has to be properly calibrated before the observed gamma ray spectra can be analyzed. This calibration is usually done by recording gamma ray spectra from various standard radioactive sources whose gamma ray energies are well known.

3.2 Semiconductor Radiation Detector

The development of semiconductor radiation detectors during the last decade has completely revolutionized the field of nuclear radiation detector. In a semiconductor detector ionizing radiation produces ion pairs which are collected by the electric field applied externally, and the detector gives an electric pulse which is proportional to the energy of ionizing radiation. The semiconductor detector have many definite advantages over the gas filled or scintillation detector . Some of these advantages are.

- i) Smaller, compact and convenient size,
- ii) Fast (few nanoseconds) rise time of output pulses.
- iii) Linear response over wide energy range.
- iv) Excellent energy resolution.

3.2.1 semiconductor Property

Let us briefly summarize the relevant properties of semiconductors before discussing the construction of semiconductor detector. According to the band theory of solids the allowed

electron energy levels in a semiconductor are peaked into bands. The conduction band lies above the valance band and is separated from it by a forbidden energy gap E_g of about $\approx 1\text{eV}$

The valance band is completely full while the conduction band is completely empty at 0°K . At higher temperature electrons from valance band get excited in to the conduction band. Their number being governed by Boltzman factor $\exp(-E_g/KT)$. If a charged particle passes through a pure semiconductor it will have to spend some energy to lift an electron from the valance band to the conduction band because no levels are allowed in the forbidden energy gap.

For semiconductor detector, instead of using pure semiconducting material (say of germanium or silicon) crystal doped with small amount of known impurity are used. For example, an atom of silicon has four valance electrons and for silicon the conduction band lies about 1.1eV above the valance band. If traces of phosphorous are added to silicon, then phosphorous having five valance electrons give up four electrons for forming bonds. While the fifth electron occupies an impurity level near the conduction band. The phosphorous impurity is called the donors(it donates electrons) and the doped silicon is now called n-type silicon. in similar way, addition of boron which has three valance electrons, results in lifting of one of silicon valance electrons to the boron atom. Such an electron occupies a level to the valance band leaving a hole behind. Here boron is called an acceptor and boron-doped silicon is called p-type silicon. In n-type silicon electrical conduction occurs by the excess electrons while in p-type silicon the vacancies or holes migrate and this gives rise to electrical conduction. In the n-type material electrons are majority carriers while in p-type holes are majority carriers.

Under the application of electric field the carriers will move across the crystal with a velocity v which is proportional to the applied electric field E or $v = \mu E$, where μ is called the mobility of the carrier. electron and holes have different mobilities. The electrons and holes moving in a crystal are affected by the impurities in two ways

- 1) *trapping* ; in this process an electron or holes gets trapped at the impurity center and after some time if get back in to the band this process takes some time and usually the trapped carrier is not able to contribute to the pulse.
- 2) *Recombination*; this is another process by which carriers are removed through the recombination of electrons and holes . however carriers which are able to escape such losses contribute to the electrical pulse formed after the ionizing events. The phenomena of recombination and trapping determine the carriers lifetime.

3.2.2 Construction of Semiconductor Detector

Basic Considerations A desirable solid state radiation detector should consist of a perfect crystal sandwiched between two conducting electrodes and an electric field applied across the electrodes, passage of nuclear radiation should produce hole-electron pairs which should move towards the electrodes so that charge is collected to signal the detection of radiation. The crystal used should have certain desirable properties:

- (a) The average energy required to produce a hole -electron pair should be as small as possible . This consideration would favor material with small energy gaps.
- (b) The material should not contain many trapping centers because the holes and electrons should be free to move all the way to the collecting electrodes. The distance covered by a carrier is determined by its mobility life time product. the important desirable property of the material is that it should have a good mobility -lifetime product for holes as well as for electrons so that they can be efficiently collected by the electrodes.
- (c) The material should have a high atomic number so that it can stop nuclear radiation more efficiently.

There are two materials which meet these desirable properties, one is silicon and the other germanium, both these material are semiconductors and they are readily available in a state of high purity and crystalline perfection. Their properties are well known and their technology is well developed. In spite of the fact that silicon and germanium are not the most ideal materials, they have been used in the construction of semiconductor detectors by cleverly doping them with proper type of impurity. We shall now outline the basic features of some commonly used semiconductor detectors.

3.2.3 Germanium Gamma Ray Detector

Lithium -drifted germanium [Ge(Li)] detectors are more suitable than silicon detectors for the detection of electromagnetic radiation. It may be recalled that the photoelectric absorption cross section for gamma rays is proportional to Z^5 and therefore germanium ($Z= 32$) is more efficient than silicon ($Z= 14$) for detection of gamma rays.

A Ge(Li) detector consists of an (n-i-p) junction device prepared by a careful drifting process. The drifted ions of lithium are effective in compensating or neutralizing the impurities in germanium and they give rise to a region of intrinsic or high resistivity material.

The intrinsic region forms the active volume of the detector where ionizing radiation (gamma rays) create hole-electron pairs which are collected by external electric field. The

Ge(Li) detector is always maintained in low temperature environment to keep up the intrinsic character of germanium. In practice, the low temperature environment is maintained by a cryostat and liquid nitrogen (77°k) dewar which together with the Ge(Li) detector form a complete detector system. Today complete Ge (Li) detector system offering large sizes (>20 cm³) and high energy resolution are commercially available. It may be added that the nuclear electronics associated with the semiconductor radiation detectors has to be more sophisticated as compared to other detectors.

3.2.4 Ge (Li) gamma ray spectrometer

The development of lithium drifted germanium [Ge(Li)] detectors during the last decade has ushered a new era in gamma ray spectroscopy.

The energy resolution of Ge(Li) spectrometers is 10- 20 times better than that of NaI(Tl) detectors. recently, large Ge(Li) detector (> 30cm³) have become commercially available and their photo peak efficiencies are almost comparable to that of medium sized used to detect X-rays and gamma rays up to ~ 20 Mev energies.

3.2.5 HPGe gamma -ray detector

General consideration

The simple junction and surface barrier detectors find widespread application for detection of alpha particles and other short-range radiations but are not easily adaptable for application that involve more penetrating radiations. their major limitation is the maximum depletion depth or active volume that can be created. using silicon or germanium of normal semiconductor purity, depletion depth beyond 2 or 3cm are difficult to achieve despite applying bias voltages that are near the breakdown level. Much greater thickness are required for the detectors intended for gamma -ray spectroscopy, which are the topic of this note. The thickness of the depletion region is given by: ?

$$d = \left(\frac{2\epsilon v}{eN} \right)^{1/2} \quad (3.1)$$

Where v is the reverse bias voltage and N is the net impurity concentration in the bulk semiconductor material (ϵ is the dielectric constant and e is the electric charge) at a given applied voltage, greater depletion depth can be only be achieved by lowering the value of N through further reduction in the net impurity concentration.

There are two general approaches that can be taken to accomplish this goal. the first is to seek further refining techniques capable of reducing the impurity concentration to approximately 10^{10} atom/cm³. The second approach to reducing net impurity concentration

is to create a compensated material in which the residual impurities are balanced by an equal concentration of dopant atoms of the opposite type.

configuration of germanium detector

- A) High-purity germanium(HPGe) detector fabrication. Techniques for the production of ultrapure germanium with impurity level as low as 10^{10} atoms/cm³ first were developed in the mid 1970s. The starting material is bulk germanium intended for use in the semiconductor industry. This material, already of high purity, is further processed using techniques of zone refining. The impurity levels are progressively reduced by locally heating the material and slowly passing a melted zone from one end of the sample to the other
- B) Since impurities tend to be more soluble in the molten germanium than in the solid, impurities are preferentially transferred to the molten zone and are swept from the sample. after many repetitions of this process, impurity levels as low as 10^9 atoms/cm³ have been achieved. The resulting germanium is perhaps the most highly purified and completely analyzed material of any kind that has ever been produced in commercial volume. Large single crystals of germanium are then slowly grown from this purified feed stock.
- C) Coaxial configuration. In this type, one electrode is fabricated at the outer cylindrical surface of a long germanium cylindrical crystal. A second cylindrical contact is provided by removing the core of the crystal and placing a contact over the inner cylindrical surface. Because the crystal can be made long in axial direction, much larger active volumes can be produced (up to 750cm³ at the time of this writing). An added advantage of coaxial geometry is that, by using a small inner diameter, larger-volume detector can be fabricated with lower capacitance than would be possible using planar geometry.

A closed-ended coaxial configuration is one in which only part of the central core is removed and the outer electrode is extended over one flat end of the cylindrical crystal. In coaxial geometry, the rectifying contact that forms the semiconductor junction can in principle be placed either at the inner or outer surface of the crystal. The electric field conditions that result are quite different.

Germanium Detector Operational Characteristics

- A) Detector cryostat and dewar. Because of the small band gap (0.7 eV), room-temperature operation of germanium detectors of any type is impossible because of the larger

thermally-induced leakage current that would result. Instead, germanium detectors must be cooled to reduce the leakage current to the point that the associated noise does not spoil their excellent energy resolution. Normally, the temperature is reduced to 77°k through the use of an insulated dewar in which a reservoir of liquid nitrogen is kept in thermal contact with the detector.

For Ge(Li) detectors, the low temperature must be maintained continuously to prevent a catastrophic redistribution of the drifted lithium that will rapidly take place at room temperature. Lithium drifting is eliminated in HPGe detectors, and they can be allowed to warm to room temperature between uses.

Germanium detectors also are universally fitted with an interlock that prevents application of high voltage to the detectors unless it has reached a low temperature. This interlock is necessary since any inadvertent application of high voltage at room temperature leads to excessively high leakage current that will likely destroy the input FET of the preamplifier. Because the FET is normally mounted inside the cryostat for cooling.

The preamplifier is normally incorporated as part of the cryostat package in modern HPGe system. It is always advantageous to locate the preamplifier as near the detector as possible to minimize capacitance. The input stages of the preamplifier normally are also cooled along with the detector to reduce electronic noise.

- B) **Energy Resolution** The dominant characteristic of germanium detectors is their excellent energy resolution when applied to γ -ray spectroscopy. The greater superiority of the germanium system in energy resolution allows the separation of many closely spaced γ -ray energies, which remain unresolved in NaI(Tl) spectrum. consequently, virtually all gamma-ray spectroscopy that involves complex energy spectra is now carried out with germanium detectors.

3.2.6 Lead shielding

The detector is placed in a lead shielding to be protected against external radiation which comes mainly from the omnipresent natural uranium and thorium and their decay products from other radioactive samples handled in the laboratory and unfortunately also from occasional contamination. The lead itself and also other construction materials contain low amount of uranium and radioactive lead. The distance between detector and lead wall should be at least 15-20 cm to minimize backscattering of gamma radiation from the walls to the detector.

Sample Holders

A sample holder is necessary to fix the samples in a defined position and distance from the deflector. If the activity of the sample high it is necessary to increase the distance between sample and deflector. But if the activity of the sample are weak, it is better short distance.

The sample holder should be made from low atomic number material to avoid scattering and absorption of gamma rays and the production of bremsstrahlung. Thus, sample containers should be constructed from poly ethylene or similar light weight plastic materials and have thin walls.

3.3 Electronics And Data Acquisition System

In modern gamma ray spectroscopy the electronics and data acquisition system consists of a detector bias supply, preamplifier, amplifier, multi-channel analyzer (MCA), a data storage device and a display for monitoring the progress of a measurement. A Schematic diagram of a modern gamma ray spectroscopy system is shown in Fig(?) and the main elements of this system are described in the following sections.

3.3.1 Detector Bias Supply

In order to collect the charges formed in the detector, a bias voltage must be applied across the detector. This voltage is chosen low enough to avoid break down or arcing, but otherwise as high as is safe since the charge collection process improves with increasing voltage. The bias voltage must be stable in order to maintain the same voltage gradients in the detector and there by the same charge collection characteristics. The optimum bias voltage for a given detector is generally specified by the manufacturer and could be from a few hundred volts for a small detector to over 4 KV for a large one depending on the detector material itself.

3.3.2 Preamplifier

A preamplifier serves a dual purpose. Firstly, it amplifies the small detector pulses by about 100 for semiconductor and gas-filled detectors, but only by unity for scintillation detectors. Secondly, by providing a match between the high impedance of the detector and the low impedance of the cable to amplifier it overcomes signal attenuation when it is required to drive a signal in to a long cable which leads to the main amplifier with only a negligible loss of amplitude. The preamplifier is usually located close to the detector to reduce the capacitance of the leads which can degrade the rise time and lower the effective signal size.

3.3.3 Amplifier

The heart of any spectroscopy system is the amplifier. For energy spectroscopy, the primary purpose of the amplifier is to magnify the amplitude of the pulses from the preamplifier from the mV range into the 0.1 to 10 V range. This is done by using the gain controls of the amplifier.

As a result of the long exponential decay on the preamplifier output pulse, there is a noticeable undershoot (overshoot) as the amplifier pulse attempts to return to the base line. At medium to high counting rates, a large fraction of the preamplifier output pulses will fall on the undershoot (overshoot) from a previous pulse. Consequently, the apparent pulse amplitudes measured for these pulses will be too low (high), which leads to a broadening of the peaks recorded in the spectrum. It is therefore essential that amplifiers incorporate a mechanism to eliminate the problems associated with the presence of these undershoots (overshoot) effects. This is achieved through the use of the so-called pole-zero cancellation method whose benefit is improved peak shapes and better energy resolution.

3.3.4 Multi-channel analyzer (MCA)

The basic task in any spectrometry is the measurement of the pulse-height distribution at the amplifier output. The data acquisition is accomplished by means of a single channel analyzer (SCA) or a multi-channel analyzer (MCA). The single channel analyzer has lower and upper discriminator levels, and produces an output logic pulse when the input pulse lies between the discriminator levels. With this device all voltage pulses in a specific range can be selected and counted. An additional SCA can be added for a second voltage range, and so on. The single channel analyzer can be used for pulse height measurements but one has to gradually search through the pulse height distribution by setting the discriminator to a narrow voltage range and going through a range of voltages since in this case only pulses whose amplitudes fall between two pre-set limits will produce the desired output. This process is time consuming, practically when high energy resolution is needed in the spectrum, because when examining one pulse height range all the remaining information has to be rejected. A way out of the difficulty mentioned above is to employ a multi-channel analyzer which can examine each pulse height and store information about the number of pulses within narrow ranges of pulse height. A pulse range of e. g. . 0-8 volts can be divided into 256, 512, 1024, up to 16 K numbers of equal pulse height intervals that are called channels.

Analog-to-Digital Converter (ADC)

The heart of such a device is the analog-to-digital converter (ADC) that measures the maximum amplitude of an analog pulse and converts that value to a digital number which is a proportional representation of the analog signal. This number is then used to select an address in a memory bank and the address contents are then incremented by one to record that pulse. In this way, sequentially arriving pulses can be sorted out and stored in the corresponding channels according to their heights. A live display of the pulse height spectrum being collected is provided via a video monitor and printer.

3.4 Dead Time

In nearly all detector systems, there will be a minimum amount of time that must separate two events in order that they be recorded as two separate pulses. In some cases the limiting time may be set by processes in the detector itself and in other cases the limit may arise in the associated electronics. This minimum time separation is usually called the dead time of the counting system. Because of the random nature of radioactive decay there is always some probability that a true event will be lost because it occurs too quickly following a preceding event. These "dead time losses" can become rather severe when high counting rates are encountered and any accurate counting measurements made under these conditions must include some correction for these losses.

3.5 Live Times

Multichannel analyzers of any size are equipped with one or several clocks. One clock records the real time along with all other data required for the evaluation of the spectra. Another clock measures the counting time and stops the measurement after a preselected period. This clock is able to compensate counting losses which occur if pulses arrive at the ADC while it is processing another pulse and not able to accept a new one. The clock is stopped as long as the ADC is "dead" that is it measures only the live time and it is therefore called a live time. If one preselects a measuring time of 1000s then the real time elapsed at the end of measurement may be 1100s, which is the sum of dead time and live time.

The ratio dead time /live time in the previous example 10% depends on the counting rate and is indicated with a panel meter at the ADC.

3.6 Calibration of the gamma-ray Spectrometer

For most measurements required in activation analysis it is useful and if absolute methods are to be applied it is necessary that the spectrometer be calibrated with respect to the energy scale and the peak counting efficiencies. This is done by measuring calibrated radio nuclide source with well-known gamma ray energies and gamma ray emission rates. These nuclide should be long lived so that they can be used for a long period. One can use a mixture of nuclide emitting decay scheme emitting many gamma rays of different energies distributed over the energy range of interest. Some useful nuclide of the first type are ^{54}Mn , ^{57}Co , ^{60}Co , ^{88}Y , ^{109}Cd , ^{137}Cs and ^{241}Am .

3.7 Evaluation Of Gamma-Ray Spectra

When the measurement of a gamma-ray spectrum is finished, it is stored in the memory of MCA as a set of data consisting of the channel numbers and the number of events recorded in each channel. The spectrum consists of a number of peaks situated on a background.

The gamma-ray spectra from the sample must be evaluated in order to find out the energies and the intensities of the gamma lines, to identify the radionuclide and to calculate their decay rates. In order to calculate the results of activation analysis, the decay rates are used as the basis for determining the amount of elements present in the sample. The table shown below gives a survey of the steps required for the evaluation of gamma-ray spectra. (Erditmann and Petri (1983))

Table 3.1: Steps for the evaluation of gamma ray spectra

step No.	Steps of the Procedure	Data required
1	Search for the peaks	
2	Determination of peak situation	
3	Calculation of gamma-ray energies	Parameters of energy calibration curve
4	Calculation of peak areas	
5	Calculation of gamma-ray emission rates	Gamma ray energies found, Parameter of efficiency calibration curve and Measuring time
6	Identification of the radionuclide	Nuclide data (gamma -ray energies and Abundance ; half life, production mode)
7	Calculation of radio nuclide decay rate	Nuclide data (gamma -ray energies and Abundance ; half life, production mode)and decay time
8	Calculation of amount of elements	Irradiation time, Neutron flux and cross-section or result from reference sample

Chapter 4

EXPERIMENT

The study was performed by taking chemical compounds of known composition, and rock samples. The chemical compounds were obtained from the Nuclear science laboratory of the physics department and the chemistry department. The two rock samples used in this work were obtained from the department of earth sciences in the faculty of science.

This chapter describes the samples used, the experimental setup, the data acquisition procedures.

4.1 Sampling and Sample Preparation

The chemical compounds are selected by their suitability for NAA.

- | | |
|------------------------------------|------------------------------------|
| a) Copper (Cu) | e) Potassium Iodate KIO_3 |
| b) Iodine (I) | f) Rock sample 1 (Code E138) |
| c) Copper sulphate CuSO_4 | g) Rock sample 2 (Code E139) |
| d) Potassium Iodide KI | |

Rock samples were obtained from the earth science department. These rocks were brought from the northern part of Ethiopia, especially in the area of Lima Limo. The major elements found in the rocks identified by inductively coupled plasma atomic spectrometry (ICP-AES) at CRPG, Nancy, France and the results were published in the Journal of Geological Society, London (Ayalew and Yirgu, 2003).

The rock samples were prepared in a powder form after grinding and sieving (in compliance with the procedures manual (HASL-300, 1997)). 30.54 gm of E38 and 8.4 gm mass of E39 samples were powdered and sealed in a suitable container for irradiation. The chemical compounds used in this work were in powder form and were irradiated as they are. Full description of the samples is shown in table 4.1.

Table 4.1: Description of the samples used in this work

Sample Code	Sample Description	mass	Activated Isotope				t_{irr}
			Symb.	Natural Abund.	Th. neutron X-section	($t_{1/2}$)	
Sm1a	Pure Copper(Cu)	4.54g	^{63}Cu ^{64}Cu	69.1% 39.9%	4.5 barn 2.2 barn	$^{64}\text{Cu} = 12.7\text{hr}$ $^{66}\text{Cu}=5\text{min}$	52.5hrs
Sm2a	Copper sulphate (CuSO4)	3.22g	Cu				52.5hrs
Sm1b	Pure Copper (Cu)		Cu				52.5hrs
Sm2b	Copper Cpd (CuSO4)		Cu				52.5hrs
Sm3a	Pure Iodine (I)	5.29g	^{127}I	100%	6.2(barn)	$^{128}\text{I}=25\text{min}$	2hrs
Sm4a	Iodine Cpd (KIO3)	4.96g	I			$^{128}\text{I}=25\text{min}$	2hrs
Sm3b	Pure Iodine (I)	5.29g	I				2hrs
Sm4b	Iodine Cpd (KIO3)	4.96g	I				2hrs
Sm5a	Pure Iodine (I)	5.06g	I				2.15hrs
Sm6a	Iodine Cpd (KI)	5.14g	I				15.5hrs
Sm5b	Pure Iodine (I)	5.06g	I				5.15hr
Sm6b	Iodine Cpd (KI)	5.14g	I				15.5hrs
Sm7	Ca(MnO3)2	6.2g	^{55}Mn	100%	13.3 (barn)	$^{56}\text{Mn}=2.577\text{hr}$	20hrs
Sm8	Rock (E38)	30.54g	^{23}Na	100%	0.528(barn)	$^{24}\text{Na}=14.96\text{hr}$	52hrs
Sm9	Rock (E39)	8.4g	^{23}Na	100%	0.528(barn)	$^{24}\text{Na}=14.96\text{hr}$	52hrs

4.2 Experimental Setup

The basic parts of the experimental set up are the HPGe detector, the electronics plus the data storage system and the neutron source.

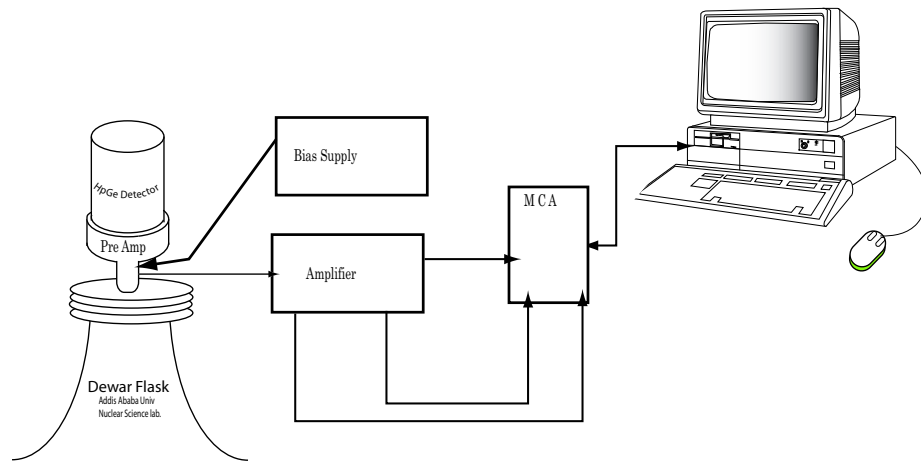


Figure 4.1: Block diagram of the experimental set up for γ -ray measurements.

Fig 4.1 shows a block diagram of the experimental set up for γ -ray measurements.

4.2.1 The Coaxial Germanium Detector

The coaxial germanium detector is basically a cylinder of germanium with an n-type contact on the outer surface, and a p-type contact on the surface of an axial well. The germanium has a net impurity level of around 10^{10} atoms/cm³ so that with moderate reverse bias, the entire volume between the electrodes is depleted and an electric field extends across this active region. Photon interaction with this region produces charge carriers which swept by the electric field to their collecting electrodes where a charge sensitive amplifier converts this charge into voltage pulse proportional to the energy deposited in the detector. The specification and performance data of the detector used is given in appendix A.

4.2.2 Electronics

The electronics of the spectrometer consists of a detector bias voltage supply, preamplifiers, amplifiers, analog-to-digital converter, data storage and computer. In this electronics system which is shown in fig 4.1 the pulses from Ge detector was amplified feed to a linear amplifier and through an analog-to -digital Converter to MCA and PC.

- a) **Electronics HV power supply:** The HV module is basically a dc-to-dc converter. The input to the high voltage Dc-to Dc converter is obtained from a conventional NIM power supply. The HV amplification is a function of a control voltage in which the sensing

circuit generates the control voltage to set and maintain a fixed high voltage (Tequash, 1995). In order to collect the charge formed in the detector, a bias voltage was placed across the detector. Its operating voltage depends on the size of the detector. The optimum bias voltage of the detector used for this work was 2700 V. The voltage which is applied to the detector ought not to be subjected to rapid change to save the damage of the field effect transistor of a preamplifier.

- b) **Preamplifier:** In order to minimize the electronic noise, the input stage of the preamplifier usually a field-effect transistor stage is cooled in the same manner as the detector. The main function of preamplifier is impedance transformation. That is it transforms the voltage which is developed across the small capacity at the input of the preamplifier in to approximately equal voltage across the high capacity of an out put cable. In systems requiting high total gain the preamplifier provides a gain up to as 100 in addition to impendence matching. (Knoll (2000))
- c) **Amplifier:** The model 1020 integrated Spectroscopy amplifies with its selection of shaping time constant was used. Preamplifiers powers was provided by means of a connector located on the rear panel of amplifier. The model 1020 amplifier were set as indicated below.

- Shaping time 2 ns as well as 4ns
- Coarse gain 20
- Fine gain 5.5

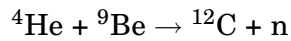
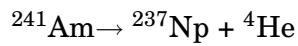
The in put polarity switch was set to match the output polarity of the preamplifier (positive).

- d) **Analog-to-Digital Converter And Data Storage:** The integrated Canberra model 1020 analog-to-digital converter was used for application in nuclear spectroscopy. The basic task in gamma spectroscopy is measurement of the pulse height distribution at the amplifier out put. The analog-to-digital converter (ADC) converters the analogy out put information of the amplifier in to a digital quantity.

The pulse height information from ADC stored on array which gives the cumulative number counts observed in each channel. The information of the pulse height was acquired using a special software (s-100 system). This programme package allows to accept the ADC out put signals, controls the counting duration, displays the data as it is taken, do simple data processing and transfer the data on request to an other device. The electronic system also determines the effective duration of the measurement. The dead time is indicated for each data taking.

4.2.3 Neutron Source

A 2 Ci Am-Be neutron source was used. In the ^{241}Am -Be source the americium-241 alloyed with beryllium, the alpha particles from americium have sufficient energy to react with beryllium which causes emission of neutrons according to the reactions.



The neutron yield of the source is approximately 77 neutrons per 10^6 alpha particles with an average energy of 4.5 MeV (Knoll, 2000). The ^{241}Am -Be source is surrounded with a paraffin wax as a moderator to thermalize the neutrons and insulated in the center of the assembly as shown in fig 4.2.

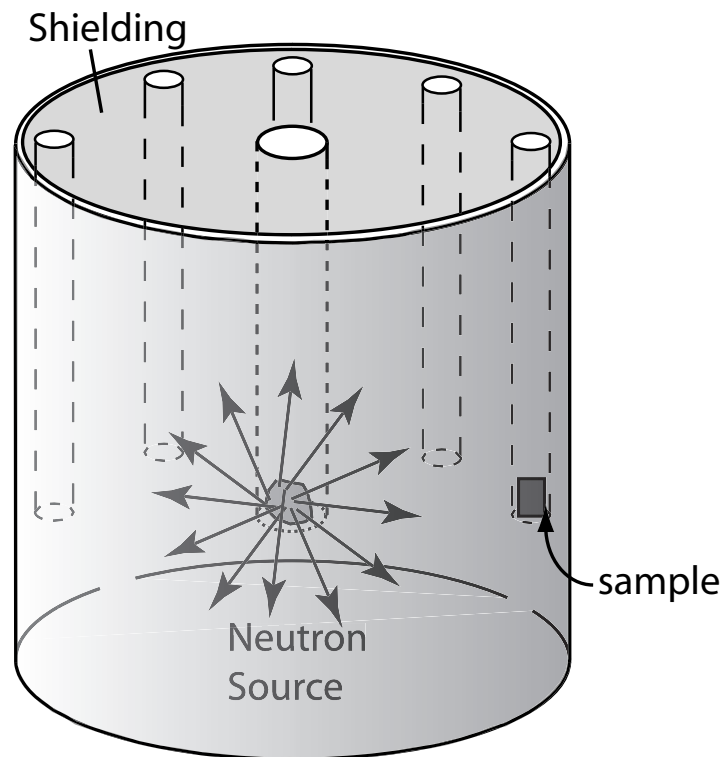


Figure 4.2: Schematic diagram of Am-Be neutron source

4.3 Experimental Procedure

First the background of the experiment room measured for 1000 seconds and the spectra for the background identified. The masses of the samples, prepared as described in the first section of this chapter, were measured using a precision balance and sealed in a sample holder for irradiation.

These samples were inserted direct into the irradiator and the time of irradiation was recorded. At the end of the pre-set irradiation time, each sample was removed from the irradiation and transferred manually to the detector and the actual duration of irradiation and delay time for measurement was recorded. The counts were recorded mostly for 1000 seconds. But for one pure iodine and KIO_3 compound the time of measurement were 500 seconds (see table 4.1).

The spectra obtained during measurement are recorded and saved as a computer file (*.MCA) where "*" stands for the sample code as appeared in table 4.1.

Chapter 5

DATA & DATA ANALYSIS

Before the measurement of activation spectra, background spectrum was measured for 1000 seconds, which is equal to measuring time. One instance of the measured background spectrum is shown in fig 5.1.

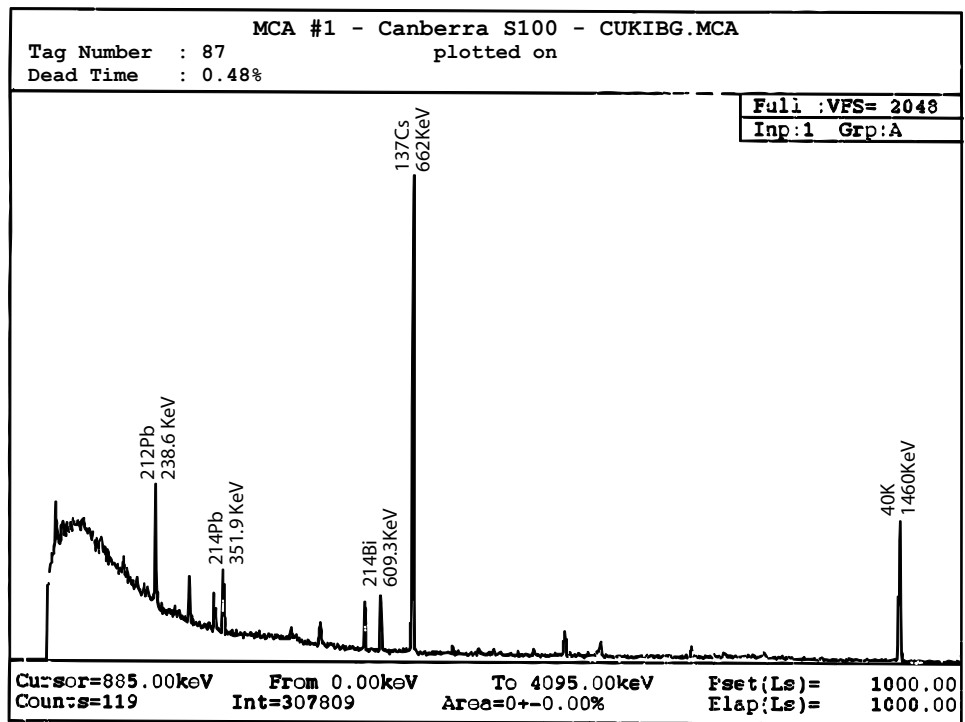


Figure 5.1: Background spectrum in the nuclear science laboratory(AAU)

The photopeaks that characterize the identified elements in the samples were located and the area under the peak determined using the inbuilt calculation mechanism in the S-100 software. The contribution of the background at the position of the peak was then subtracted and the net area under the photo peak was calculated.

The net photopeak area was used to determine the concentration of the element of interest.

5.1 Copper in Pure and Compound Form

Pure copper samples (Sm1a, Sm1b) and Copper sulphate compound CuSO_4 samples (Sm2a, Sm2b) were used in this category of samples.

The pure copper samples are used as a standard to determine the concentration in the CuSO_4 samples by the use the 511 keV gamma photon coming from annihilation of the positron emitted from the decay of neutron activated ^{63}Cu (i.e. ^{64}Cu).

Ordinary copper consists of 69% ^{63}Cu for which the neutron capture cross section is 4.5 barn and 31% ^{65}Cu for which the cross section is 2.2 barn (See table 4.1). The relative neutron absorption is therefore proportional to 69×4.5 and 31×2.2 respectively.

Of the neutrons absorbed by ^{63}Cu only 82% produce the nuclide ^{64}Cu which is of interest to us. Further more, ^{66}Cu has a half-life of only 5 minutes while ^{64}Cu has a half life of 12.7 hr hence after a few hours delayed time for measurement the ^{64}Cu lost by decay only small amount of its activity, while ^{66}Cu decayed almost completely. This is also true for ^{37}S . Since the cross-section of the $^{63}\text{Cu}(n,\gamma)^{64}\text{Cu}$ reaction is fairly large a small mass of samples could be used to have good specific activity.

The decay scheme of ^{64}Cu is represented in appendix(A) main decay modes are the electron capture and the β^- decay directly to the ground state of ^{64}Zn respectively. The 1346 KeV line from decay to Ni-64 appears only with a small branching ratio (0.5 %). Therefore we used the β^+ decay of ^{64}Cu (branching ratio 19%) to identify these in the sample. In this way two annihilation gamma ray photons of energy 511 KeV each are created. The back ground spectrum was stripped from the sample spectra. The main single dominant photo peak in the spectrum remained is the 511 KeV line.

To obtain information about the copper content in CuSO_4 we have used a comparative method utilizing sample of copper which is irradiated simultaneously with the CuSO_4 samples and assayed in the same manner. This technique of relative measurements can be carried out easily and does not required absolute assay technique.

The concentrations of the sample C_{sam} and the standard C_{std} are related by:

$$\begin{aligned} \frac{C_{\text{sam}}}{C_{\text{std}}} &= \frac{W_{\text{std}}}{W_{\text{sam}}} \left(\frac{m_{\text{sam}} (e^{-\lambda T_d})_{\text{sam}}}{m_{\text{std}} (e^{-\lambda T_d})_{\text{std}}} \right) \\ &= \frac{W_{\text{std}}}{W_{\text{sam}}} \left(\frac{A_{\text{sam}}}{A_{\text{std}}} \right) \end{aligned}$$

where C is concentration of the sample and W is weight of the sample, A is the activity concentration of the sample (sam) and standard (std), m is mass of the element, λ decay constant for the isotope and T_d is decay time.

The amount of ^{64}Cu activated in the CuSO_4 sample was determined from the observed peak content of the 511 KeV line in the CuSO_4 and copper spectra. The area of the photo

peak is given in table 6.2 (See analysis result) which is found to be (39.2%) amount of copper in the CuSO_4 sample varies only by (1.5%) from the known value of copper in CuSO_4 chemical mass ratio which is 39.8%.

The major source of error in deducing the amount of copper in CuSO_4 could be raised from several sources of errors contribute to the variation of the experimental result. These errors might be from counting statistics and background subtraction in addition to this, analytical source of errors include surface contamination of sample during sample preparation and nuclear source of errors which include self- shielding for thermal neutrons in the sample, failure due to flux gradient during irradiation, interference from competing reactions and interference from primary or secondary reaction.

The γ -spectrum contains the characters of 511 KeV for copper according to the decay scheme shown in appendix A was identified for pure copper sample and CuSO_4 samples are given in the figures shown below.

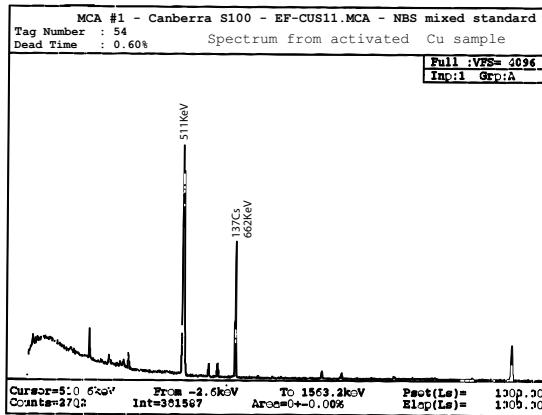


Figure 5.2: Spectrum From Cu Sample

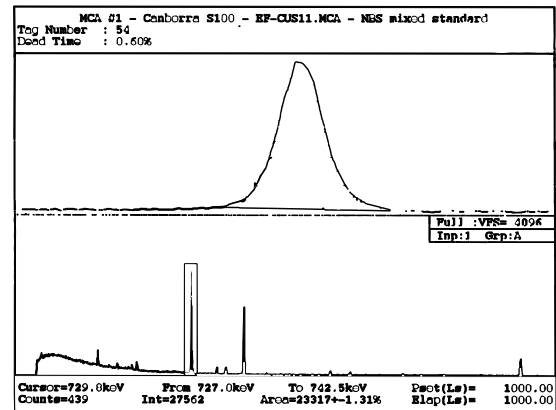


Figure 5.3: Spectrum from Cu Sample: Analysis

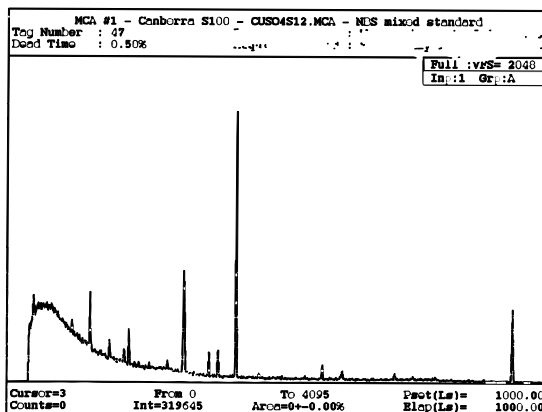


Figure 5.4: Spectrum From CuSO_4 Sample

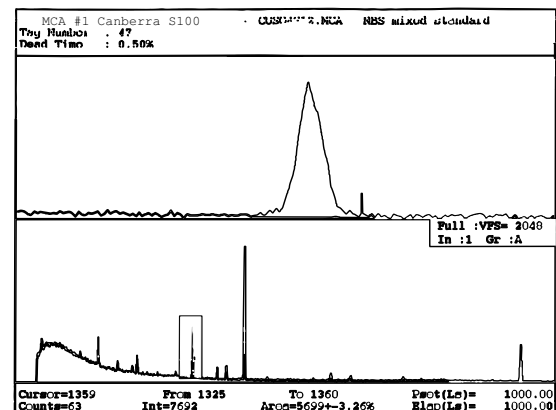


Figure 5.5: Spectrum from CuSO_4 Sample: Analysis

Table 5.1: Analysis of CuSO₄ Samples

Sample Code	Mass of Sample	Mass of Cu in the sample (g)	lambda	t _d (sec)	Counts =Activity	Expected Mass ratio	Activity Ratio	Corr. factor due to diff. in t _d	Exptl. Mass Ratio	% error	Remark
Sm1a	4.54	4.54	1.52E-05	8280	23317						
Sm2a	3.22	1.28	1.52E-05	10188	5987	0.282	0.256	1.029	0.264	0.064	Standard Sample
Sm1b	4.54	4.54	1.52E-05	12600	20604						Standard
Sm2b	3.22	1.28	1.52E-05	15012	5699	0.282	0.276	1.037	0.286	-0.016	Sample

5.2 Iodine and its Compounds

The second part of the experiment is to determine the concentration of Iodine within the KIO_4 and KI chemical compound samples. The amount of iodine in these chemical compounds was determined from the observed gamma-ray spectra by comparison with corresponding spectra of activated pure iodine by photo peak area method. The net area of the 443 KeV photo peak is directly proportional to the gamma emission rate of the corresponding ^{128}I isotope.

The spectrum due to ^{128}I at 443KeV are shown in figures 5.6 and 5.7. The comparative method which is used to find the amount of iodine in the chemical compound samples is given by using the equation(1.12).

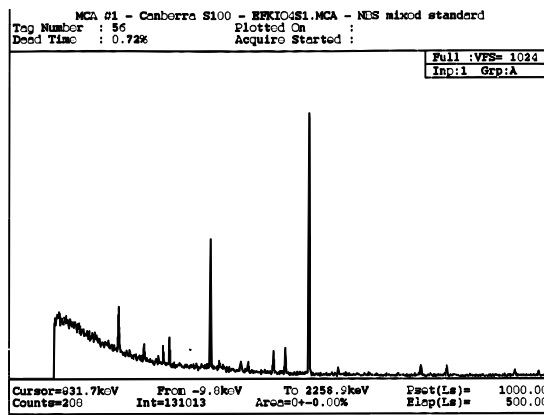


Figure 5.6: Spectrum From KIO_3 Sample ($t_d = 1018$ sec)

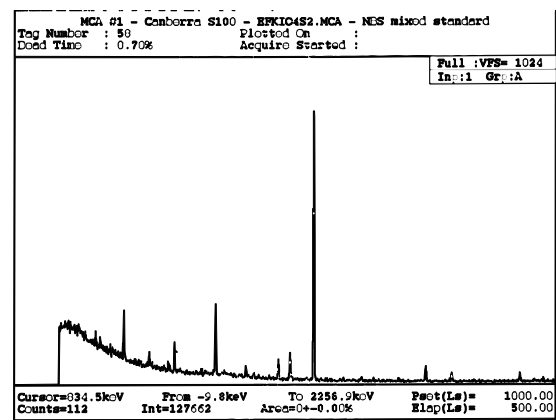


Figure 5.7: Spectrum from KIO_3 Sample: ($t_d=2376$ sec)

The average concentration of iodine in KIO_3 was 59.2% and in KI was 73.2%. The relative error with the known concentration by chemical mass ratio that is 55.6% for KIO_3 and 77.7% for KI become 6.5% and 4.6% respectively.

Since the half-life of ^{128}I only 25 minutes it decays rapidly and changing rapidly the counting rate. Due to this the relative error become high comparing with the first experiment that is copper which has relatively optimum half life (12.8 hr).

In addition to this, in the case of KI and I samples they were not irradiated for equal time and the measuring time was relatively long. Therefore, the major source of errors in this experiment arise from counting statistics. Moreover, background subtraction, self shielding for thermal neutron in the sample and contamination during the sample preparation might contribute for source of errors.

Taking into consideration the error sources; the obtained results are in good agreement with the expectation. This shows the good reliability of neutron activation analysis (NAA) for quantitative investigation of materials.

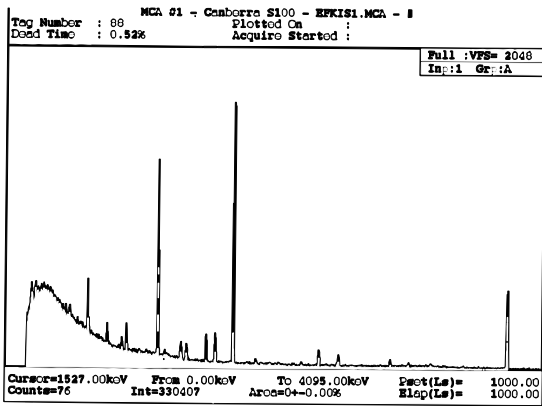


Figure 5.8: Spectrum From KI Sample ($t_d=300$ sec)

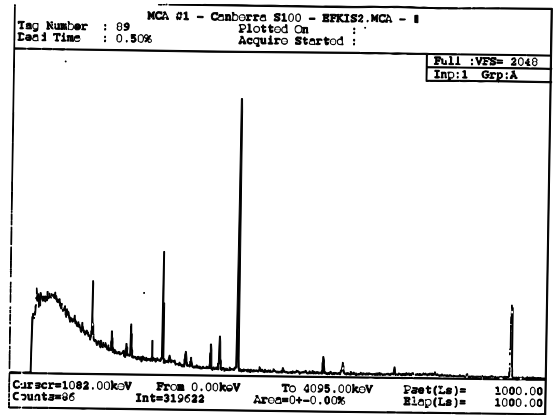


Figure 5.9: Spectrum from KI Sample ($t_d=1440$ sec)

Table 5.2: Analysis of KIO₃ Samples

Sample Code	Mass of Sample	Mass of I in the sample (g)	lambda	t _d (sec)	Counts =Activity	Expected Mass ratio	Activity Ratio	Corr. factor due to diff. in t _d	Exptl. Mass Ratio	% error	Remark
Sm3a	5.29	5.29	0.000462	300	4412						
Sm4a	4.96	2.94	0.000462	1018.8	1874	0.556	0.425	1.394	0.592	-0.06481	Standard Sample
Sm3b	5.29	5.29	0.000462	1800	2183						Standard Sample
Sm4b	4.96	2.94	0.000462	2376	988	0.556	0.455	1.308	0.591	-0.06216	Standard Sample

Table 5.3: Analysis of Potassium Iodide (KI)

Sample Code	Mass of Sample	Mass of I in the sample (g)	lambda	t _d (sec)	t _{irr} sec	Counts =Activity	Expected Mass ratio	Activity Ratio	Corr. factor due to diff. in t _d	Corr. factor due to diff. in t _{irr}	Exptl. Mass Ratio	% error	Remark
Sm5a	5.06	5.06	4.62E-4	2160	7740	3156							Standard Sample
Sm6a	5.14	3.93	4.62E-4	300	55800	5436	0.777	1.722	0.423	0.972	0.709	0.087	Standard Sample
Sm5b	5.06	5.29	4.62E-4	3300	18540	1738							Standard Sample
Sm6b	5.14	3.93	4.62E-4	1440	55800	3096	0.777	1.781	0.423	0.9999	0.754	0.029	Standard Sample

5.3 Identification of Mn and Na from Samples

Mn was identified in the compound $\text{Ca}(\text{MnO}_3)_2$ (Sm7) and Na was identified from the two rock samples Rock E38 (Sm8) and Rock E39 (Sm9).

Concentration of Mn in sample 7 was not determined as there was lack of standard. The same true for Na element in the rock samples as pure Na is not suitable for such analysis. However the activity ratio as obtained in the our work corresponds with the concentration ratio of the compound containing Na in the rock samples (see table below). This verifies the validity of the measurement technique we have used in this experiment.

Table 5.4: Identification of Mn and Na

Sample Code	Mass of Sample	t(half) in sec	lambda	t _d (sec)	t _{irr} (sec)	Counts =Activity
Sm7	6.20	9277.20	7.47E-05	1800	72000	137572
Sm8	30.54	53856.00	1.29E-05	3960	187200	1170
Sm9	30.54	53856.00	1.29E-05	2880	187200	378

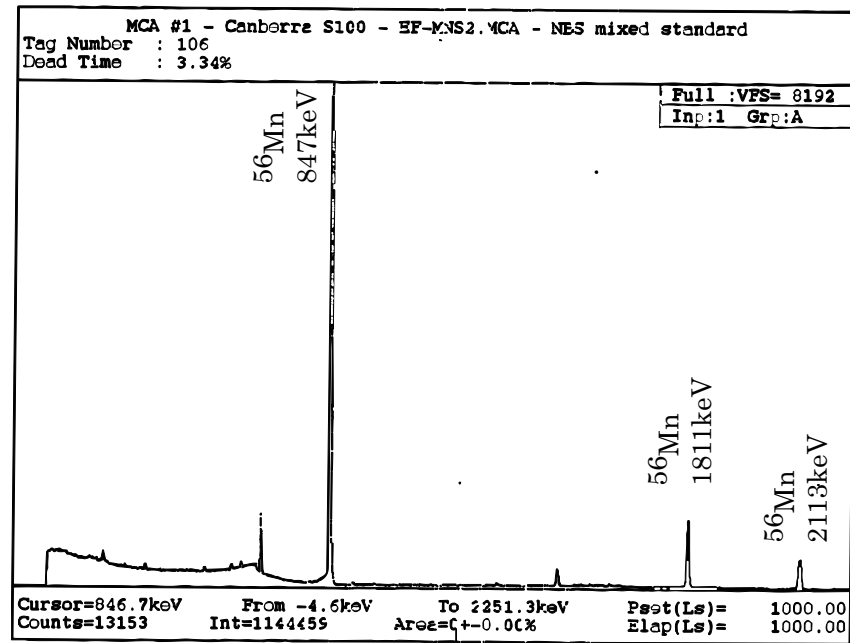


Figure 5.10: Spectrum of Mn from $\text{Ca}(\text{MnO}_3)_2$

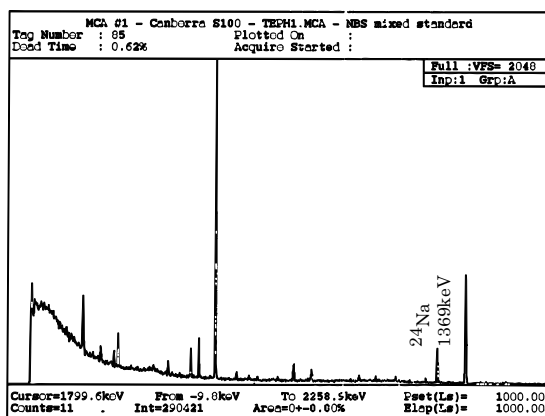


Figure 5.11: ^{24}Na Spectrum from E138 at 1369keV

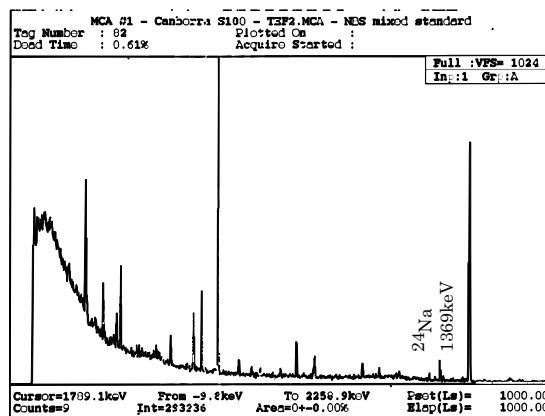


Figure 5.12: ^{24}Na Spectrum from E139 at 1369keV

5.4 Conclusion

Neutron activation analysis using the ^{242}Am -Be neutron source has proved to be useful to identify and determine the amount of a constituent element of a given sample. In this work the concentration of copper in CuSO_4 was determined. The measured values in the two instances are within -1.2 to 6.4% from the chemical mass ratio that is known to be 28.2% by weight. Similarly, the concentrations of iodine in KIO_4 and KI were determined. The relative error in these measurements were found to be 6.21% and 6.48% respectively.

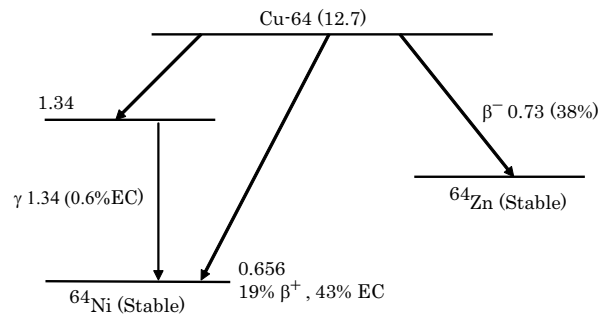
The errors reported here are within the errors that may appear due to measurement of masses and fluctuations in the background photo spectrum, in our case the detector was not shielded at all.

The results of this work shows that the Neutron Activation Analysis (NAA) technique, as it is now, can be used for environmental radioanalysis and with improved shielding and precision measurement can grow to analyse trace elements in samples of interest in medicine, forensic, mining and many other application. nuclear method for further research purpose and it is a reliable method for quantitative and identification analysis.

Appendix A

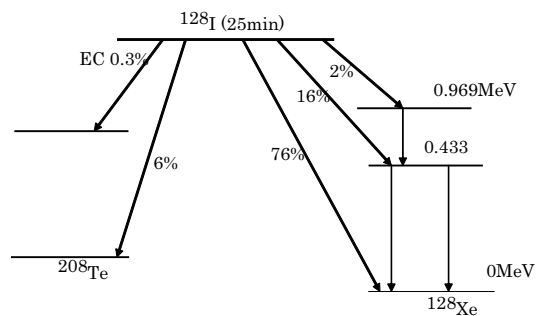
Decay Schemes

A.1 Decay Scheme of ^{64}Cu



- Production: $^{63}\text{Cu}(n,\gamma)^{64}\text{Cu}$
- Mode of decay: β^- (38%), EC (43%) and β^+ (19%)
- Main gamma energy: 511 KeV (19%), 1.34 MeV (0.6%)

A.2 Decay Scheme of ^{128}I



- Production: $^{127}\text{I}(n,\gamma)^{128}\text{I}$
- Mode of decay: β^- (93.7%), EC (6.3%)
- Main gamma energy: 443 KeV (16%), 527 KeV (1.57%), 969.4 KeV (0.4%)

Appendix B

Detector Configuration

Detector specification and performance data of HPGe detector

The coaxial germanium detector which is used for measurement of gamma-ray energy have the following properties:

1. Specification:

Relative efficiency	50%
Resolution	2 KeV (FWHM) at 1.33 MeV
Peak/Compton	62:1

2. Physical Performance Data

Geometry	Coaxial one open end; closed end facing to window
Diameter	48 mm
Length	34.5 mm
Active area facing window	18.1 cubic cm
Distance from window	5 mm

3. Electrical characterstics

Recommended bias voltage	3500 V
Bias voltage used for this work	2700 V

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DECLARATION

I the under signed declare that the thesis is my original work, has not been presented for a degree in any other university and that all sources of material used for the thesis have been duly acknowledged.

Name: _____

Signature: _____

This Thesis has been submitted for examination with my approval as university advisor

Name: _____

Signature: _____