Estimation of Amount of Sodium in the Natural Rock Sample Using Neutron Activation Analysis

By:
Tewodros Gezhagn

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Tewodros Gezhagn

Department of Physics
Addis Ababa University

Approved by Examining Board:

1. Prof. A.K.Chaubey Advisor____________

2. Dr. TilahunTesfaye Examiner____________ 26-03 /2012

3. Dr. Bhatnagar Examiner____________

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Dedication

This work is dedicated to my beloved mother
“Inspector Workuha Mamo”

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Acknowledgment

First of all I would like to thank God with his virgin marry. I would like to give my best respect and thanks to my advisor Proff. A. K. Chaubay who had built my confidence of performing this thesis work in my choice and as I desire to conduct; And even for his unexpressed behavior of treating me with simplifying the narrower things.

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Abstract

This study deals with the elemental analysis of rock sample which are collected from North Wollo (Woldia). Neutron Activation Analysis (NAA) is a sensitive analytical technique useful for performing both qualitative and quantitative multi-elemental analysis of major, minor and trace elements in samples from almost every conceivable field of scientific on technical interest. Am-Be Neutron source was used. The mass of sodium found in 2.103 gm. of irradiated sample is 0.034861216 gm.; which means 1.65 % of the irradiated sample is sodium.

1. Introduction

As far as our life is concerned we should know the very elemental contents of our naturally gifted rocks, plants, soils and everything we have in nature. One of the objective of the governmental office named Geological Survey of Ethiopia is to identify, analyze and enterprate data from geological samples such as minerals, rocks, soil, stream sediments and water etc.; even though the office do not have a full and well organized documents on the elemental contents of rock samples found through-out the country.

This study deals with the elemental analysis of rock sample which are collected from North Wollo (Woldia). I am interested in studying the elemental analysis of these rock samples not only since it is easy but also no one have done elemental analysis study in this place before and in addition to these reason, I have a great interest in playing my own role of finding the elemental contents of some rocks found in Ethiopia.

I have used Neutron Activation Analysis (NAA) which is one of the methods for qualitative of quantitative determination and identification of elements from different chemical compounds and rock samples.

1.1. Objectives

This study has the following objectives:

- Checking as the rock samples which are collected from Woldia holds sodium.
- Estimate the amount of sodium in the Natural Rock Sample Using Neutron Activation Analysis
- Recommend a wider project to find some other useful elements.

2. Theoretical Approach

2.1 Neutron Activation Analysis (NAA)

2.1.1 Introduction

Neutron Activation Analysis (NAA) is a sensitive analytical technique useful for performing both qualitative and quantitative multi-elemental analysis of major, minor and trace elements in samples from almost every conceivable field of scientific on technical interest. For many elements and applications, NAA offers sensitivities that are superior to those attainable by other methods, on the order of part per billion or better. In addition, because of its accuracy and reliability NAA is generally recognized as the “referee method” of choice when new procedures are being developed or when other methods yield results that do not agree, world wide application of NAA is so widespread, it is estimated that approximately 100,000 samples undergo analysis each year.

Neutron activation analysis was discovered in 1936 when Hevesy and Levi found that samples containing certain rare earth elements become highly radioactive after exposure to a source of neutrons. From this observation, they quickly recognized the potential of employing nuclear reactions on samples followed by measurement of the induced radioactivity to facilitate both qualitative of quantitative identification of the elements present in the sample.

The basic essentials required to carry out an analysis of sample by NAA are a source of neutrons, instrumentation suitable for detecting gamma rays, and a detailed knowledge of the reactions that occur when neutron interact with target nuclei.

When initially the sample is irradiated with neutrons in a nuclear reactor or in other neutron source, the stable nucleus absorbs one neutron and becomes a radioactive nucleus. The concentration of the stable element of interest in the sample can be measured by detecting the decay of these nuclei.

The radioactive nuclei emit characteristic gamma rays. Detection of the specific gamma rays (of specific energy) indicates presence of particular elements. Suitable semiconductor radiation detectors may be used for quantitative measurement. The concentration of various component elements in a given samples are found by computer data reduction of gamma ray spectra sequential instrumental neutron activation analysis allows quantitative measurement of up to about 35 elements in small samples of 5 to 100mg. The lower detection limit is in parts per million or parts per billion, depending on the elements. (Source 3)

2.1.2 Basic Principle

In typical NAA, when neutron interacts with the target nucleus via a non-elastic collision, a compound nucleus is due to the binding energy of the neutron with the nucleus. The compound nucleus will almost instantaneously de-excite into a more stable configuration through emission of one or more characteristic prompt gamma rays. In many cases, this new configuration yields a radioactive nucleus which also de-exits (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 nucleus. Depending up on the particular radioactive species, half-lives can range from fraction of a second to several years.

With respect to the time of measurement, in principle, NAA falls into two categories:

i) Prompt gamma-ray neutron activation analysis (PGNAA), where measurements take place during irradiation or

ii) Delayed gamma-ray neutron activation analysis (DGNAA), where the measurements follow radioactive decay.

When one mentions NAA is generally assumed that measurement of the delayed gamma rays is intended. About 70% of the elements have properties suitable for measurement by NAA.

The sequence of events that occur during the most common type of nuclear reaction used for activation analysis, namely the neutron capture or (n, gamma) reaction is shown in figure 1 below. (Source 5)

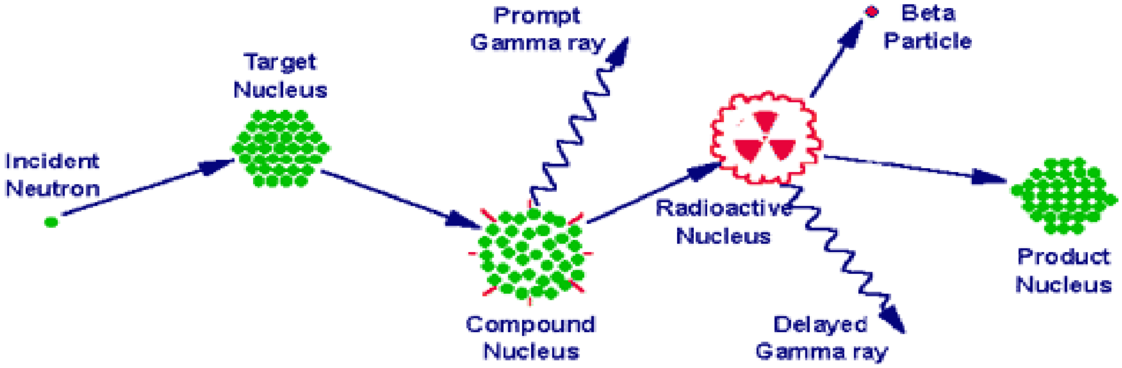


Figure 1: Sequence of events that occur during the most common type of nuclear reaction used for activation analysis (source-3)

The energies of delayed gamma rays one used to identify component sample elements. The count of gamma rays of a specific energy indicates the amount of an element in the sample.

The usual procedure involves placing of the samples to be analyzed plus a number of suitable standards into the neutron field.

The samples are then analyzed using an appropriate gamma ray detector system. On striking a suitable detector the gamma ray energies are converted to an electrical signal that is processed as a count in an energy spectrum. The accumulation of gamma counts at a particular energy generates a curve, the area of which is proportional to the radioactivity of the characteristic radionuclide. Comparing against standards allow the establishment of a relationship that can be used to determine the abundance of a particular element or elements.

The measured count rate (R) of gamma rays from the decay of a specific isotope in the irradiated sample can be related to the amount (n) of the original, stable isotope in the sample through the following equation (1)

$$R = \varepsilon I_{\gamma} A \tag{1}$$

$$R = \varepsilon I_{\gamma} n \varphi \sigma (1 - e^{-\lambda t_i}) e^{-\lambda t_d} \tag{2}$$

Where R = measured gamma – ray count rate (Cps)

ε =Absolute detector efficiency

I_{γ} = Absolute gamma ray abundance

A = Absolute activity of isotope ($^{A+1}$Z)

= radioactive decay constants(s^{-1}) for isotope $^{A+1}$Z

t_i = irradiation time (sec)

t_d = decay time (sec)

φ = Neutron flux (neutrons.cm^{-2}.sec^{-1})

n =Number of atoms of isotope ^{A}Z in sample

σ = Neutron capture cross section (cm^{2}) for isotope ^{A}Z

If the neutrons flux, neutron captures cross section, absolute detector efficiency E and absolute gamma ray abundance I_{γ} are known, the number of atoms (n) of isotope ^{A}Z in the sample can be calculated directly.

In most cases, however, a standard is irradiated and counted under similar conditions as the sample, and the mass of the element in the sample is found by comparing the measured count rate ® for the sample and standard through the following equation (z)

$$W_{sam} = W_{std} \frac{R_{sam}}{R_{std}} \qquad (3)$$

Where: W_{sam} = mass of elt in sample (g)

W_{std} = mass of elt in standard (g)

R_{sam}= sample gamma-ray count rate (cps)

R_{std} = standard gamma-ray count rate (cps)

The qualitative characteristics are the energy of the emitted gamma quanta (E) and the half-life of the nuclide (T ½). The quantitative characteristic is the intensity, which is the number of gamma quanta of energy E measured per unit time. (Source - 3)

Steps of the analysis:

Step 1: Sample preparation means in most cases only pulverizing, homogenizing, mass determination, packing, as well as the selection of the just analytical process and the preparation of the standards, if any.

Step 2: far irradiation one can choose then the various types of neutron sources according to need and availability.

Step 3: after irradiation the analysis face the dilemma whether or not a chemical separation should be carried out for better sensitivity. If it is needed, the measurement shall be made after the separation. If there is no chemical treatment, the measurement is performed after a suitable cooling time (t_c).

Step 4: Measurement, evaluation and calculation involve taking the gamma spectra and the calculating trace element concentrations of the sample. The most widely used gamma spectrometer consists of germanium based semi-conductors connected to a computer used as a multichannel analyzer far spectra evaluation and calculation. (Source - 4)

2.1.3 Advantage, Limitations and Applications of NAA

a) Advantage

NAA is a highly sensitive analytical technique and require only a minimum of sample handling and treatment before irrational.

§ As the vast majority of sample are completely transparent to both the probe (the neutron) and the analytical signal (the gamma ray) NAA is free of matrix interference.

§ Reagent or laboratory contamination is minimized because NAA does not require sample pre-treatment such as digestion or dissolution.

§ NAA enables the direct analysis of compact pieces like coins, precise stones, seed grains or other small organs from plants and animals without destruction and in many cases samples can be used for further investigations.

§ The risk of contamination and trace element loss, which are the most series source of error in trace element analysis, are minimal in the case of NAA with this method compact lump of rocks, ceramics or other hard materials with freshly broken, untouched surfaces can be directly irradiated and measured.

§ Easy far calibration with accuracy since the neutrons used for activation as well as the gamma rays used for measurement thoroughly penetrates the same materials, so that the relationship between element concentration and measured activity is nearly matrix-independent. This means, the analytical sample and reference sample need not consists of the same matrix element as required for many other methods.

§ NAA can be applied directly to a great number of different sample materials at once.

b) Limitations

Interferences can still occur when different component sample elements produce similar gamma rays. This problem can be circumvented by choosing alternative gamma rays or by counting only after the shorter – lived nuclide had decayed.

Sensitivities and accuracy depends on the concentration of the element and radionuclide parameters (i.e., parent isotope abundance, neutron cross-section, half-life, and gamma ray abundance). Element sensitivities vary from 10^{-3} to 10^{-10} grams per gram of sample.

The detection limit for a particular element depends upon the measured count rate (R) for a given isotope and consequently the signal to noise ratio, can be increased by any combination of the following:

1. Increase the detector efficiency (moving the sample closer to the detector)
2. Increase the irradiation time (t_i)
3. Decrease the decay time (t_d)

In many cases the sensitively of the measurement can be improved by increasing the measurement time t_m, which enhances the overall signal or total number of counts (R. t_m). Another common limitation of instrumental NAA is caused by the back matrix. When it produces a large background that masks the signal and interest, the signal to noise ratio becomes very low and the detection limit is reduced extensively. Time delays or chemical separations may be used to minimize this effect.

c) Application

Advances in semiconductor detector systems coupled with improvements in nuclear reactions have widened the scope of the neutron activation analysis.

Neutron activation analysis has been used successfully in research as diversified as nutrient uptake in plants, petro-genetic modeling of geologic phenomena, identification of importees in new metals and alloys, environmental monitoring, and analysis of forensic evidence for criminal and civil cases.

Small samples (as small as a few micrograms) of 30 to 35 major, minor and trace elements may be routinely analyzed by instrumental neutron activation analysis. Radio chemical neutron activation analysis techniques, although destructive to the sample, enhance the number of determinable elements to about 50. Some scientific, engineering, and industrial applications of neutron activation analysis are listed below in table 1.

Agriculture	Bee pulp, liquids, hay, oil, fish
Archeology/Anthropology	Ceramic utensils, obsidian, teeth, bone
Biology	Chemicals, sugar, enzymes, solutions, ants
Botany	Wheat spores
Chemistry	Oxides, alts, pure crystals, and metals
Engineering & Industry	Pure metals, chemical compounds, oils, thin film deposits plastic films, alloy, rocks
Fisheries	Fish, shells
Forestry	Wood, phloem, tree needless, soil
Geology	Rocks, meteorites, moon samples, gems, minerals
Medicine	Water, skin, hair, nails, alternative medicine
Oceanography	Fossil, sediments, basalts
Pharmacy	Chemicals
Forensic	Bullets, paint, glass, metals, gunshot residue swabs

Table (1) – Some application of NAA (Source - 3)

A simple example of neutron activation analysis involves the measurement of iridium in soils. The ^{252}Cf(a neutron emitter) isotope is used for neutron activation analysis, to inspect airline luggage for hidden explosives, to gauge the moisture content of soil and other materials, in bare hole legging in geology, and in human cervix concern therapy.

Estimation of precious metals such as gold and silver and coinage metals is popular. Some rare studies include lipid study; a mobile analysis system, using prompt gamma neutron activation analysis, for the non-destructive identification of chemical warfare agents and explosives in different kinds of ammunition and trace elements in 52 Chinese medicines.

Neutron scattering has proved to be a valuable tool for studying the molecular structure and motion of molecules of interest to manufacturing and life process. Accelerator and reactors produce low-speed neutron with wavelength appropriate to ‘see’ structures of the size of magnetic microstructures of the size of magnetic microstructures and DNA molecules. Neutrons can penetrate deeply into bulk materials and use their magnetic moment or strong interaction forces to preferentially scatter from magnetic domains or hydrogen atoms is long chain nucleosomes. Based on their reflectivity properties, neutrons are also used in materials surface and interface studies.

Landmine detection and radioisotope power generation are identified as possible future applications of NAA. (Source - 3)

2.1.4 Sampling Methods

The aim of sampling is to take a representative sample of an object to be characterized. It must be representative at least with respect to the properties that are to be measured. In analytical chemistry that means the chemical composition of the sample elements, compounds, phases or whatever 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 homogenous amount of material.

From sampling 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.

2.1.4.1 Sample size and shape

The amount of sample to be 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 circumstance even a few micrograms will be sufficient for trace element determination.

Virtually any solid material can be analyzed by this method, but there are geometric constraints – the sample must file into the irradiation vial and should present a consistent geometry, for example, finely ground powders or well 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.

2.1.4.2 Sample preparation

Once a representative sample has been identified from the material to be characterized, it is usually necessarily to take sub samples and to prepare them for irradiation in the neutron source. Sample preparation steps include drying, aching or pre-concentration of one or few elements may become necessary if the elements to be measured. (Source -2)

2.2 Neutron sources

Three main sources of neutrons are: (1) radioactive sources; (2) research nuclear reactor (nuclear reactors used as neutron sources); and (3) ion and electron accelerators including neutron generators. Research nuclear reactors have the highest neutron fluxes, but are limited concerning on-site determination, price, and availability. Consequently, nuclear reactors are used predominantly for delayed γ-neutron activation analysis of very minute amounts or for sensitive neutron radiography. However, it is used also for PGNAA. When site irradiation is important, neutron generators or radioactive sources are used.

2.2.1 Radioactive Neutron Sources

Radionuclide neutron sources are composed either of a radionuclide emitting α- or γ-rays, together with an appropriate surrounding material, or from a radionuclide decaying by spontaneous fission. The γ-emitter in photo neutron sources is surrounded by beryllium or deuterium (such as D_2O) and neutrons are emitted due to the (γ, n) reaction:

$$\gamma + {}^{9}\mathrm{Be} \longrightarrow 2\alpha + n - 1.67\ \mathrm{MeV} \qquad \gamma + \mathrm{D} \longrightarrow \mathrm{H} + n - 2.23\ \mathrm{MeV}$$

Very few radionuclides with reasonable half-lives emit -rays, with energies that high. Due to this, together with the disadvantage of the long range of -rays, the use of that kind of neutron source is very limited. The most commonly used photo neutrons source is ^{124}Sb-Be, which emits neutrons of 26 1.5 eV. ^{124}Sb is produced by reactor irradiation of natural antimony. ^{124}Sb emits several γ-rays, including 1.692-MeV photons (with intensity of 48%), and has half-life of 60.9 d. The neutron source is composed of two parts, the core (a sphere or a cylinder) made of irradiated antimony metal and a shell of beryllium metal about 2 cm thick. The practical yield is about 10 n/s per I Ci^{124}Sb.

Most neutron sources use an α- interaction with ^{9}Be:

$$^{9}\mathrm{Be} + \alpha \longrightarrow {}^{12}\mathrm{C} + n + 5.91\ \mathrm{MeV}\ ({}^{12}\mathrm{C}\text{ is in the 4.43-MeV excited state})$$

The spectra of these neutrons spread up to 10 to 12 MeV. The common α-emitters used Ra, Po, Pu, and Am. The main properties of the sources are summarized in Table 1.

Table 2 Properties of Neuron Sources from Be (α, n) C Reaction

A emitter ⟶	^{210}Po	^{239}Pu	^{241}Am
Neutron yield (n s^{-1} Ci^{-1})	$2.5 * 10^6$	$1.7 * 10^6$	$2.2 * 10^6$
Half-life	138.4 d	24,360 yr.	458 yr.
Approximate size ($cm^3 Ci^{-1}$)	0.1	12	3
Heating ($mWCi^{-1}$)	32	31	33
γ −dose (rad h^{-1} Ci^{-1})	0.11 (4.43 MeV)	0.08 (4.43 MeV)	10(60 eV), 0.1(4.43 MeV)

The Po-Be and Am-Be sources are prepared by mixing fine powders of beryllium with polonium metal or americium oxide. The Pu-Be source is an intermetallic compound, Pu-Be. The mixture or the intermetallic compound is doubly encapsulated, first in an inner capsule of tantalum and then in an outer capsule of stainless steel. In 80% of the (α, n) interactions, the ^{12}C nucleus is left in the 4.43-MeV exited state, which decays with an emission of 4.43 MeV γ-photons. The γ-dose of these three ^{9}Be (α, n) ^{14}C sources is lower than with the initially used source of Ra-Be due to the γ-rays from the Ra source. Lower doses of γ-rays are associated with the neutron source of spontaneous fission. The main source of spontaneous fission is ^{252}Cf.

$$\mathbf{^{252}Cf \xrightarrow[t_{1/2}=2.65\,yr]{spontaneous\ fission} two\ fission\ products + 3.8n + 200MeV}$$

The half-life for spontaneous fission is 85.5 years, and for alpha emission is 2.73 years, and the effective half-life is 2.65 years:

$$\frac{1}{t_{eff}} = \frac{1}{t_{\alpha}} + \frac{1}{t_{sf}} \qquad \text{………………………..} \quad 4$$

The ^{252}Cf sources are very compact. The neutron spectrum is very similar to that of neutron-induced fission, with a mean energy of 2.348 MeV. The neutron yield is 2.31 X 10^6 n/s. In order to increase the flux of neutrons, it is cheaper to surround the ^{252}Cf with U than to use larger amounts of Cf. These devices are called neutron multipliers. ^{252}Cf (1 mg) combined with 1.4 g U (93.4%) and polyethylene as moderator is equivalent to a neutron source containing 33 mg ^{252}Cf. This device has thermal and fast neutron fluxes of 4 X 10^8 and 6X 10^8 n/s cm^2.(Source -6)

2.2.2 Research Nuclear Reactor

Research nuclear reactors are usually large devices in which fissionable material, almost exclusively U, is fissioned into two nuclides with simultaneous emission of neutrons that induce further fissions in a chain reaction. The fission-produced neutrons are very energetic. The cross-section for neutron-induced fission of fissionable nuclides increases with decreasing energy of the neutrons; in order to increase the neutron activity, moderators that slowed down the neutrons are added to the reactor. To reflect back some of the neutrons that leaked from the reactor core, reflectors are used. The fission process releases large amounts of energy, mainly due to the stopping of the recoiling two fissioned particles, and the system is cooled by a coolant (either liquid or gas). The nuclear reactors are categorized according to their fuel, moderator, coolant, reflector, and configuration. Almost all research nuclear reactors (neutron sources) are heterogeneous reactors in which the fuel is in the form of rods. The fuel is enriched (natural uranium has only 0.7% the fissile material). Most research reactors have 93 to 99%. Many of the reactors have rods that are ^{235}U-Al alloys; however, some of the newer designs (mainly those converted to 20% ^{235}U) are of the uranium-silicide type. TRIGA reactors operate with uranium-zirconium hydride fuel that, due to its large negative temperature coefficient of reactivity, allow the operation of the reactor in pulses. In the Light Water Reactor (LWR), ordinary water (H_2O) is used both as a moderator and as a coolant. The reflector is mainly graphite, but there are also Be or H_2O reflected reactors. The construction is either pool type or tank-in-pool type. Due to the relatively high cross-section for capturing thermal neutrons by H atoms, the flux of neutrons is LWRs always contains large fractions of fast and epithermal neutrons. The available power is in the range of 10 to 5000 kW, with neutron fluxes of $5 * 10^{14}$ to $1.5 * 10^{18}$ n/m^2.s. The neutrons are usually divided into three groups according to their energy: (1) thermal energy with most expected energy of kT = 0.025 eV at room temperature; this group is usually extended up to the Cd (thermal neutrons absorber) cut off of 0.5 eV (2) neutrons with energy between 0.5 eV and 1 MeV are called epithermal neutrons; and (3) neutrons with energies above 1 MeV are called fast neutrons. Many reactors are unique in their design; however, there are some commercial types that are more common—the American TRIGA and the Canadian Slowpoke. The TRIGA reactor is a popular multipurpose research reactor. About 50 of them are operating with power levels of 18 kW to 3 MW (fluxes of $7 * 10^{15}$ to $3 * 10^{17}$ n/m^2.s).). The most common types are those of 250 kW and 1 MW. They are of the pool type, graphite reflected with uranium-zirconium hydride fuel, and enrichment of 10 to 70%. The Slowpoke reactor is a low-power (20 kW) reactor designed specifically as a teaching aid, with additional purposes of activation analysis and production of small amounts of radioisotopes. The system is designed to operate remotely. It can be provided with up to five irradiation sites in the core with a flux of 10 n/m^2 s and five further tubes outsides the reflector with half that flux. Heavy-water research reactors are tank types. They usually have enriched uranium fuel, heavy-water moderator and cooled, and heavy-water and graphite reflected. Due to the low cross-section for thermal neutrons, absorption by D and O, they are characterized by well-thermalized neutron flux (very little epithermal and fast neutrons fluxes except inside the core). Due to the lower moderation power of D compared to H, the physical size of heavy water reactors is larger and hence they have a large available irradiation volume. Their power is usually between 10 and 26 MW (fluxes of up to 2 X 10^{18} n/m^2. s).(Source-6)

2.2.3 Neutrons from Accelerators and Neutron Generators

Charged particle accelerators can produce neutron fluxes by (d,n), (p,n), or (α,n) reactions. Electron accelerating machines can produce neutron fluxes via (γ,n) reactions, where the -flux is obtained by stopping the electrons in a high-Z material. The main reactions for producing neutron fluxes are D (d,n)^3 He, T(d,n)^{4}He, and ^{9}Be(d,n)^{10}BThe first two reactions are exothermic and require very little acceleration of the deuteron beams; this is the reason why they are used in the neutron generators, which are very small accelerators with accelerations of 150 to 500 keV. The disadvantage is that the target material is gas (D_2 or T_2) adsorbed onto metal. If the target is heated, too much of the gas will be desorbed from the metal target; this limits the current of the bombarding deuteron beam and consequently the neutron flux. In the case where a higher flux of neutrons is required, a target of Be is used; however, the deuterons should have an energy of a few MeV. The most common use of the reaction T(d,n) He is due to the low acceleration needed and the higher cross-section of this reaction, as compared to D(d,n)He. Different high-voltage generators are used in various neutron generators: Cockroft-Walton, insulating core transformer (ICT), Van de Graaff, and electrostatic rotor machines. A deuteron beam is produced by various ion sources and accelerated to one to few hundred keV. The beam may also be a mixture of 50% tritium and 50% deuterium. Typical beam intensities are of the order of a few milliamps, but intensities as high as 150 mA have been used. Neutrons of 14 MeV are produced from the interaction of the beam with a large surface-tritiated target. The target consists of a few hundred microns of titanium, zirconium, or palladium evaporated on a 30-mm diameter backing disk and saturated with tritium gas. The disk is water cooled and made of heat-conducting material, silver, or copper. Rotating targets are sometimes used to ensure heat dissipation.

Commercially compact sealed-tube neutron generators are also available. The usual neutron generators have fluxes up to 5 X 10^{11} n/s; however, special generators with outputs of 5 X 10^{13} ns^{-1} were also constructed. (Source - 6)

2.3 Radiation Interaction

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

Charged particle radiation: Fast electrons and heavy 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 radiations 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.

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.

2.3.1 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: Interaction of Photons with matter

Kinds of interaction	Effects of interaction
1) Interaction with atomic electrons 2) Interaction with nucleons 3) Interaction with the electric field 4) Interaction with the meson field	a) Complete absorption b) Elastic scattering c) Interaction with the electric 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). (Source - 2)

2.3.1.1 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 hole and cannot 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 f the atom. The photoelectron appears with an energy given by

$$E_e = hv - E_b \tag{5}$$

E_b is the binding energy of the electron and hv 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 Ey and Z, but a rough approximation is: (Knoll, 2000).

$$\tau \cong constant * \frac{Z^n}{E_\gamma^{3.5}} \tag{6}$$

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. (Source - 2)

2.3.1.2 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 θ = 0) to maximum energy E_m (when θ = π).

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 $\theta = \pi$. (Source - 2)

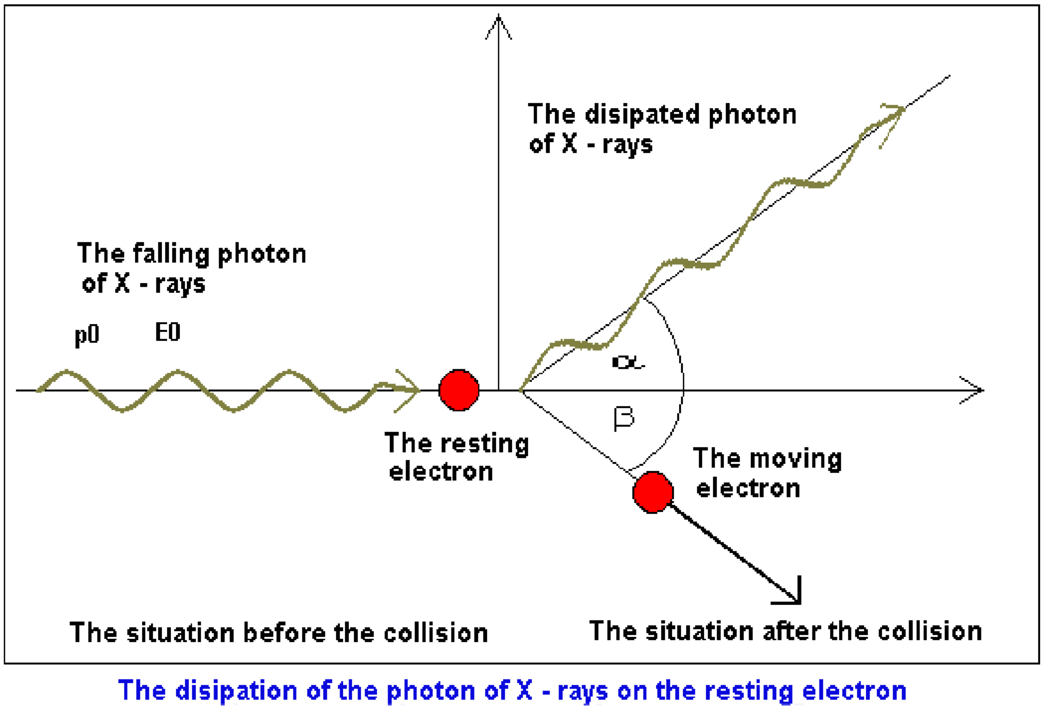


Figure 2: Events in the Compton scattering process.(Source - 8)

2.3.1.3 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 hv.

$$hv = (T_{-} + m_{o}C^{c}) + (T_{+} + m_{o}C^{c}) \tag{7}$$

Where $T\_$ and T_{+} are the kinetic energies of the electron and positron respectively and m_oC^2 = 0.511 MeV is the electronic rest mass energy. The process occurs only in the field 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:

$$probability_{(pp)} = k\,(\log(hv))(Z)^2 \tag{8}$$

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 energy of 0.511 MeV equal to the rest energy of an electron.

$$e^{+} + e^{-} \longrightarrow 2hv \tag{9}$$

The two gamma-rays in equation (9) 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 radiations.

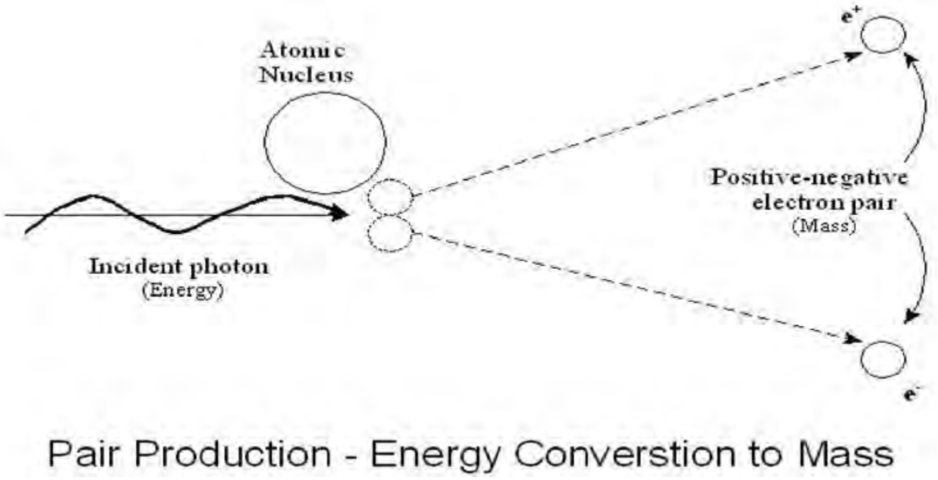


Figure 3: Pair production (Source – 7)

2.3.2 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.

2.3.2.1 Energy Classification

For the purpose of study of neutron interactions; neutrons are classified as below:

- 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. (Source - 2)

2.3.2.2 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,) 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. **Non-elastic scattering** (Nuclear Reactions Involving Emission of Charged Particles) Non-elastic reactions occur at high neutron energies [(n,x), (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 N with slow neutrons:

$$^{14}_{7}N + ^{1}_{0}n \longrightarrow ^{16}_{6}C + ^{1}_{1}H$$

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 irradiative capture process. Conditions for such reactions are especially favorable during slow neutron (with energy < 1 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. (Source - 2)

2.3.2.3. 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 neuron 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 in to two parts according to the probabilities of the two reactions.

$$\sigma_t = \sigma_s + \sigma_{abs} \qquad (10)$$

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

$$\sigma_{abs} = \sigma(\mathrm{n},\gamma) + \sigma_{(\mathrm{n},\rho)} + \sigma_{(\mathrm{n},2\mathrm{n})} + \sigma_{(\mathrm{n},\mathrm{n})} \qquad (11)$$

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

$$\sigma(\mathrm{n},\gamma) = \sigma(\mathrm{n},\gamma)g + \sigma(\mathrm{n},\gamma)m1 + \sigma(\mathrm{n},\gamma)m2 + \sigma(\mathrm{n},\gamma)m3 \ldots.e\,t\,c. \qquad (12)$$

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 cannot be correlated with the area of the atomic nuclei. They are reaction constants with the dimension of an area (Erditmann and Petri, 1983).

$$R = \frac{-dn}{dt} = \gamma\sigma N \qquad (13)$$

Where R is the reaction rate N is the number of the target atoms and ø 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 neutrons stays with in the sphere of influence of an atomic nucleus for a long time and its probability of reacting is greater than that a fast neutron. Indeed it is found that at low energies the reaction cross sections for most nuclide are inversely proportional to the velocity of the neutron.

The thermal (n, γ) cross-section vary by large factors from to isotope and thus from element to element. Also, γ-ray emission probabilities and half-lives can vary by large factors between radionuclides. Therefore, in the sensitivity of NAA varies strongly with element involved. Some elements, in particular light ones cannot be detected by thermal NAA (i.e. H, C, N, O, Si, P, S and Ca). (Source - 2)

2.4 Kinetics of activation

In the case of nuclear reaction induced by neutrons the radioactivity of the examined isotope depends on the flux of the neutrons and the cross section of the given reaction. The cross section and neutron flux highly depend on the energy of neutrons, and therefore the usual activation is

$$\mathrm{R} = \mathrm{N}\int_0^{\infty} \sigma(E).\varphi(E)dE \tag{14}$$

Where: N = number of interacting isotopes
$\sigma(\boldsymbol{E})$ = Cross section [in cm^2] at neutron energy of E [in eV]
R= reaction rate

In nuclear reactors the integral in equation (14) is usually replaced by the sum of two integrals separating the thermal and epithermal regions, the lower limit of the epithermal component of a neutron spectrum most commonly is 0.55 eV:

$$\mathrm{R} = \mathrm{N}(\Phi_{\mathrm{th}}\sigma_{\mathrm{th}} + \Phi_{\mathrm{e}}.\mathrm{I_o}) \tag{15}$$

σ_{th}: Conventional thermal neutron flux [in cm^2]

φ_{th}: Effective thermal neutron cross-section [in cm^2]

ϕ_e: Conventional epithermal neutron flux [in $cm^{-2}s^{-1}eV^{-1}$]

I_o: Resonance integral cross section (in epithermal region), for 1/E epithermal spectrum [in cm^2]

The activity (A) of the isotopes depends on time. During irradiation the activity of the radioactive isotope produced grows according to a saturation characteristic governed by a saturation factor, $S = 1 - e^{-\lambda t_i}$. Subsequent to the irradiation the isotope decays according to the exponential law:$D = e^{-\lambda t_d}$: Where t_i = time of irradiation; t_d: time of decay;   : decay constant.

$$A = (\varphi_{th}\sigma_{th} + \varphi_e I_o)\frac{m f_i N_{Av}}{A_{rel}} S.D \tag{16}$$

Where:

A_{rel}: Atomic mass of target element
f_i: Isotopic abundance
m: The mass of irradiated element
N_{Av}: Avogadro number

The intensity of the measured gamma line is proportional to the activity. The measured parameter is the total energy peak area (N_p) at a particular energy given by:

$$N_p = A f_\gamma \varepsilon_\gamma t_m \tag{17}$$

The efficiency (ε_γ) of a semiconductor detector varies with gamma energy. The emission probability of gamma photon at a given energy is thef_γ, t_m is the measuring time. (Source -4)

2.5 Methods of standardization

For many evaluations of y-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.

2.5.1 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 for an irradiation period of tirr then at the end of irradiation the activity or decay rate A of a radio nuclide built up from (Erditmann and Petri, 1983):

$$A_o = N_o \frac{ma}{M} \Phi_{th} \sigma_{th} [1 - \exp(-\lambda t_i)] \qquad (18)$$

Where, N is Avogadro's number, M is the atomic weight of the element, a is the abundance of the isotope yielding the ratio nuclide to be measured, is the thermal cross section is the decay constant of the radio nuclide.

The activity after a waiting period td, 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, A can be calculated as:

$$A_o = \frac{p\lambda exp(\lambda t_d)}{h\eta[1 - \exp(-\lambda t_m)]} \qquad (19)$$

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

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

$$1 - \exp(-\lambda t_i) = \text{B (build-up term)} \qquad (20)$$

$$\frac{\lambda exp(\lambda t_d)}{1 - exp(-\lambda t_m)} = \text{D (decay and measuring term} \qquad (21)$$

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

$$\frac{exp(\lambda t_d)}{t_m} = D \qquad (22)$$

If all entities are accurately known the expected, activity and the measured activity are equal and the amount of element can be calculated:

$$m = \frac{PMD}{N_o a h \eta \phi_{th} \sigma_{th} B} \qquad (23)$$

The calculation of analytical result from equation (6) is called the absolute method. A 20-50% uncertainty was to be expected if analysis were carried out by this method using equation (5) this was due to the fact that tabulated data for gamma-ray abundance and reaction cross-section from different sources were used. (Source - 2)

2.5.2 Classic 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 values 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).

$$m_{i,s} = m_{i,r}\frac{A_{o,s}}{A_{o,r}} \tag{24}$$

Where $m_{i,s}$ and $m_{i,r}$ are the amount of element in the sample and in the reference sample respectively. The Ao’s are the decay rates at some reference point of time; usually the end of the irradiation period is chosen. From the peak areas Ps and Pr 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:

$$\frac{A_{o,s}}{A_{o,r}} = \frac{P_s D_s B_r}{P_r D_r B_s} \tag{25}$$

Where: B and D correspond to Equation 18 and 19 or 20. 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. (Source – 2)

2.6 Measurement and evaluation

The modern gamma ray measuring systems consists of gamma detector, usually a HPGe type and sometimes NaI (TI) Scintillation crystals. The detectors are connected to a multichannel analyzer (MCA) by an appropriate electronic system (preamplifier, spectroscopy amplifier, etc). Nowadays, the MCAs are computer based systems with the ability of an automatic spectrum evaluation. The most important parameters of the detectors are the efficiency and resolution. The efficiency of the NaI (Tl) detectors is duite high, but their resolution is poor comparing to the semiconductor germanium detector. The typical resolution of a 3x3” NaI(Tl) detector at 1333 kev energy is 90 KeV and the same for a HPGe detector with a 30% relative efficiency (related to a 3x3” NaI(Tl) in similar circumstances) is 1.9 – 2.2 KeV. (Source - 4)

2.6.1 Analysis of the gamma spectra

The usual objective of the measurements by gamma ray spectrometers is the determination of the number and energy of the photons emitted by the source. The peak location and the peak area in the spectra have to be determined. The peak location is a measure of the gamma energy, while the peak area is proportional to the photon emission rate. For the energy measurement the pulse height scale must be calibrated with standard sources emitting photons of known energies. In order to be determined by using sources of known activities.

For the determination of the peak areas the background under the peak interval has to be subtracted. The net count (N_p) results from $N_p = N_{int} – N_B$, (N_{int} integral under the peak and N_B refers to the background). The peak area can also be calculated by computer programs which fit an analytical function to the peak. The shape is described basically by a Gaussian function, modified by suitable auxiliary functions. Thus all the peaks including also multiplets can be automatically analyzed. (Source - 4)

2.6.2 Counting Statistics

The nuclear decay processes occur at random, and follows a poisson distribution, where the standard deviation (σ) equals to $N^{1/2}$ (N is the observed number of events). In gamma spectrometry, the peak area is the measured parameter. The standard deviation is:
$\sigma = (N + 2N_B)^{1/2}$: Where the confidence level is 68%.

The counting statistic is only one of the possible sources of errors in NAA, the overall value depending on a number of different factors (e.g. sample preparation, weighting, and uncertainty of standardization). (Source -4)

2.7 Radiation Detector

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 output 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 detects 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, has 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 y 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, y 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. (Source - 2)

2.7.1 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. Before we describe the operations of a scintillation detector, we will see how scintillation will be produced. (Source - 2)

2.7.1.1 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 photopherescence. 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. (Source - 2)

2.7.1.2 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 scintillation can dissipate all its energy in it, 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.
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 scintillators are NaI (Ti). We will see deeply about NaI (Ti) 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 photo cathode 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 107-108 by the time electrons reach the last stage which is called the anod. In this way the initial ionizing radiation given rise to a burst of electrons at the anode where an electrical pulse is taken out from 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-cesium) on the inner surface of the flat top of the tube envelope. The equality 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. (Source - 2)

2.7.1.3 Scintillation gamma rays spectrometer

One of the most efficient methods 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 a aluminum 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. (Source - 2)

2.7.2 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 has 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.

2.7.2.1 Semiconductor Property

Let us briefly summarize the relevant properties of semiconductors before discussion 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 Eg of about ~ 1ev.

The valance band is completely full while the conduction band is completely empty at 0^0 k. At higher temperature electrons from valance band get excited in to the conduction band. Their number being governed by Boltzmann factor exp (Eg/KT). If a charged particle passes through a pure semiconductor it will have to spend some energy to life an electron from the valence 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 valence electrons and for silicon the conduction band lies about 1.lev 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 levels 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 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 = uE, where u is called the mobility of the carrier, electron and holes have different mobility’s. 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. (Source - 2)

2.7.2.2 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 doing them with proper type of impurity. We shall now outline the basic features of some commonly used semiconductor detectors. (Source - 2)

2.7.2.3 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) creates 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 (77^0k) Dewar which is 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. (Source - 2)

2.7.2.4 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(Ti) detectors. Recently, large Ge(Li) detector (> $30cm^3$) have become commercially available and their photo peak efficiencies are almost comparable to that of medium sized used to detector X-rays and gamma rays up to ~ 20 Mev energies.

2.7.2.5 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:

$$d = \left(\frac{2\varepsilon v}{eN}\right)^{1/2} \tag{26}$$

Where v is the reverse bias voltage and N is the net impurity concentration in the bulk semiconductor material (€ 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 farther 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/cm^3. 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.

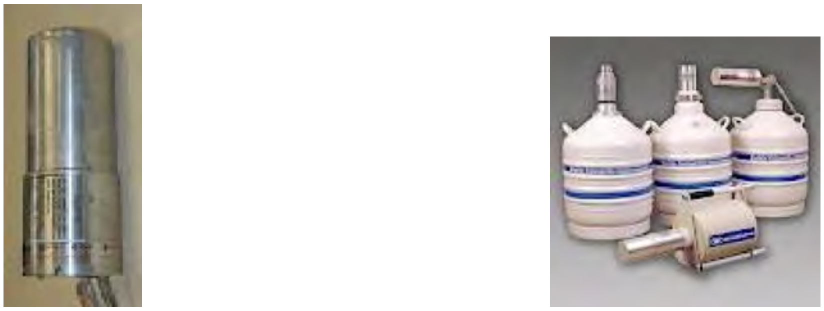


Figure 4: a) HPGe detector b) HPGe detector inserted in liquid nitrogen (Source -9)

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/cm^3 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/cm^3 have been achieved. The resulting germanium is perhaps the most highly purified and completely analyzed material f 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 77k 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 y-ray spectroscopy. The greater superiority of the germanium system in energy resolution allows the separation of many closely spaced y-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.

2.7.2.6 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 defector. If the activity of the sample high it is necessary to increase the distance between sample and defector. But if the activity of the sample is 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.

2.8 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 photography of a modern gamma ray spectroscopy system is shown in Fig(5) and the main elements of this system are described in the following sections.

2.8.1 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.

2.8.2 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.

2.8.3 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 under shoot (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 cancelation method whose benefit is improved peak shapes and better energy resolution.

2.8.4 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 need 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 pulses 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.

2.9 Dead Time

In nearly all detector systems, there will be a minimum amount of time that must separate two events in order that they are recorded as two separate pulses. In some cases the limiting time may be a set by processed 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 tether severe when high counting rate are encountered and any accurate counting measurements made under these conditions must include some correction for these losses.

2.10 Live Times

Multichannel analyzers of any size are equipped with one or several clocks. One clock records the real time day of 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 lessees which occur 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 times. 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/live time in the previous example 10% depends on the counting rate and is indicated with a panel meter at the ADC. (Source - 2)

2.11 Calibration of the gamma-ray Spectrometer

For most measurements required in activation 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 canting efficiencies. This is done by measuring calibrated radio nuclide source with well-known gamma ray energies and gamma ray emission rates. This 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 nuclides of the first type are ^{54}Mn, ^{57}Co, ^{60}Co, ^{88}y, ^{109}cd, ^{137}Cs and ^{241}Am.

2.12 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. (Source - 2)

Table 3.1: Steps for the evaluation of gamma ray spectra

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

3. Experimental Approach

3.1 – possible types of experiment

There are two types of possible experiments which can be takes placed in NAA. These are; In beam or ON-time experiments and Off-line or Off beam experiments.

3.1.1 In beam (ON-time) experiment: Beam of neutrons is also telling on the sample, and compound nucleus is decaying by emission of prompt γ-radiations of high energy which can be detected by a pair spectrometer or large size NaI (Te) spectrometer. The measurement is takes placed during irradiation time.

3.1.2 Off-line /0ff-Beam Experiment: After the emission of prompt $\gamma -$ radiation we get from the ground state of compound nucleus which may be radioactive and may decay by emission of α , β radiation which populate excited state of radioactive daughter product, so by counting α or β particle or by counting low energy of daughter product ,one can estimate the amount of new-element found.

This work is possible in any lab where the requirements of source and detectors are complete. Only those elements whose parent nucleus is stable and natural abundance is large are possible to be studied. Also the next isotope produced must be radioactive with measurable half-life; neither too short nor too long; and the decay scheme of radioactive nuclei produced must be well known. (Source – Lecture note given by Proff. A. K. Chaubey)

3.2 Objectives of the experiment

The main objectives of the experiment are:

- prepare the sample to be irradiated
- irradiate the sample using Am-Be neutron source
- Study the Gaussian curve generated by the accumulation of gamma at a particular energy from the detector.

3.3 Experimental procedure

The experimental procedure which is used until having the energy spectrum from the studied sample is:

1. The rock samples are collected from the study area which is Woldia (North Wollo).
2. The sample is prepared by hammering to make it in powder form for irradiation.
3. The powdered sample is prepared by putting it inside the sample holder.
4. The mass of the sample is measured before having the sample in to the neutron source.
5. Two iodines of known masses are placed side by side to the sample and plastered together.
6. The sample which is prepared in powder form and the two iodines are placed inside the “Am-Be” neutron source for fifteen says after being plastered.
7. The detector is prepared by filling the liquid nitrogen and setting the computer for detection of the radiation.
8. The irradiated sample and the two iodines are placed on the detector every next time alone and the detector starts counting after setting the counting time.

3.4 Sample collection

The samples to be studied were collected from the mountains found in North Wollo, Woldia. Weldiya (also spelled Woldia or Woldiya) is a hillside market town, capital of the Semien Wollo Zone, and woreda in northern Ethiopia. Located north of Dessie and southeast of Lalibela in the Amhara Region, this town has a latitude and longitude of 11°50′N 39°36′E / 11.833°N 39.6°E and an elevation of 2112 meters above sea level according to the global positioning system (GPS). During the sample collection, representative sampling were taken as the main criteria, hence, the corresponding site of collection should have representative samples.

Even though representative sampling were taken as the main criteria, Rocks with unusual looks were the main target of studding and while collecting f these unusually locked sample s, great care were not taken for contamination. (Source - 10)

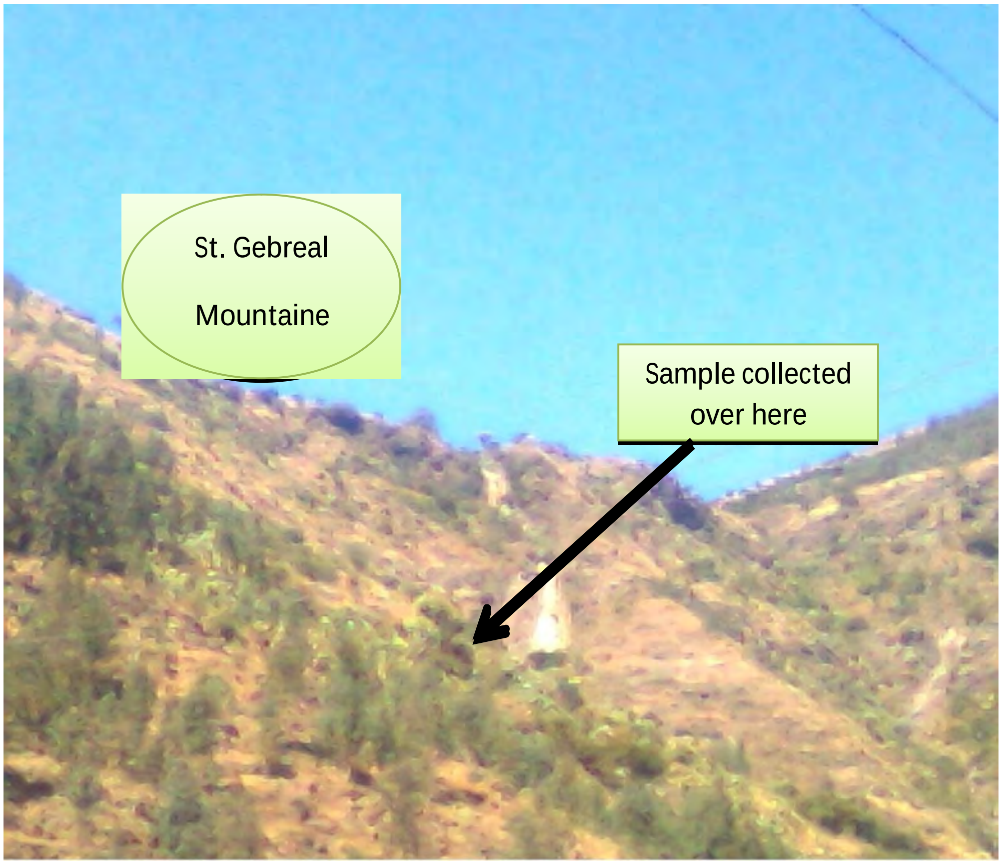


Figure -5- The area on which the Sample is collected

3.5 Sample preparation

The representative samples which are collected were expected to be prepared in order to have it in to the neutron source.

First, the samples were hammered (grinded) by using stone (black) after covering the samples by papers in order to live contamination and after making the covering. Samples to have a powder form, small amount of samples were placed inside the sample holder and it plastered after measuring the mass of the sample, with two Iodines of different sample. Since the main aim of sample preparation is to put the sample to be studied in to the neutron source , the plastered sample were attached to the plastic with length of around 30cm and inserted into the" Am-Be" neutron source.

3.6 Am-Be neutron source

A 2ci Am-Be Neutron source was used. In the 241 Am-Be source the americium-241 alleged with Beryllium, the alpha particles from americium have sufficient energy to react with Beryllium which causes emission of neutrons according to the reactions.

$$241_{Am} \text{ à } 237_{Np} + 4He \qquad (27)$$

$$4He + 9Be \text{ à } 12_{C} + n \qquad (28)$$

The neutron yield of the source is approximately 77 neutrons per 10^{6} alpha particles with an average energy of 4.5 ?. The 241-Am-Be source is surrounded with a paraffin wax as a moderator to thermalize the neutrons and insulted in the center of the assembly as slow in fig 4

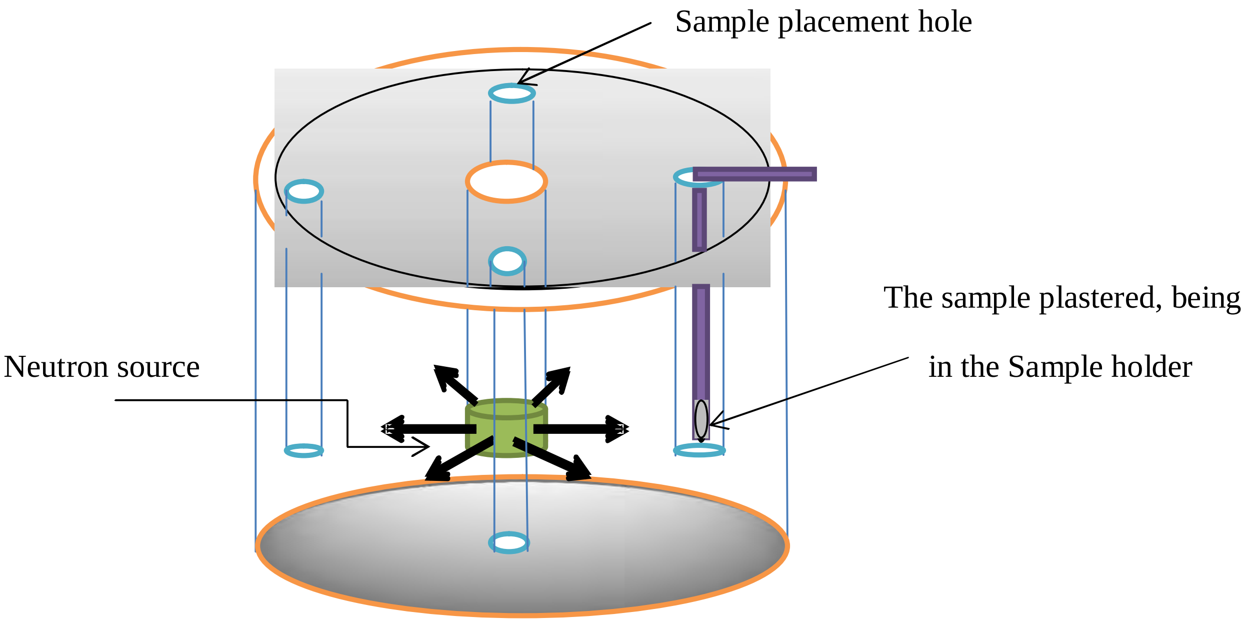


Figure 6: Am – Be neutron source (Source - 2)

3.7 Irradiation of sample

After having the sample prepared and the neutron source, it is time to have the irradiation of the sample for some specified time. Hence, the thesis work is expected to be completed in one semester and also there are many researchers who use the Am-Be neutron sources, the sample had no much time to be irradiated rather only for about 15 days. This shortage of time had a great effect on the study of the possible elements found inside the sample hence, depends on the half-life of the elements determined and the elements determined and the amounts of elements present. The sample will be irradiated for an arbitrary time, if the sample is completely unknown, and then for proper isotope identification, the sample should be irradiated again for a time that will provide enough activity.

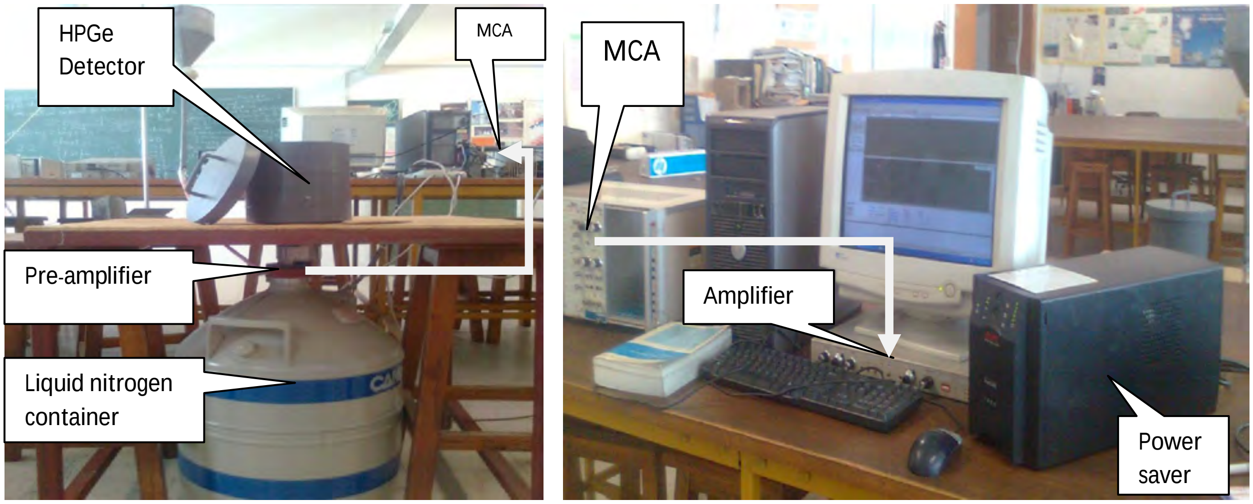
A 2.103 gm. of rock sample and two standard of iodine of mass 0.6787gm and 0.7816 gm. were inserted in to the neutron source and irradiated for about 15 days for activation.

After the irradiation, the irradiated sample is brought to the high purity Germanium detector looking for γ rays. The position of each peak determines the energy of the gamma ray and the area under the peak is proportional to the concentration.

3.8 experimental Set-up

The experimental set-up on the applied gamma ray spectrometer consists of HpGe detector with its electronic circuits: detector bias voltage supply, preamplifier, amplifier, Analog-to-digital converter, multichannel analyzer and computer. The measurements were performed and analyzed using Genie 2000 software.

The set-up is given in figure-3 (a) and (b); the two figures are from front and back side respectively.



(a) (b)

Figure – 7 – The gamma ray measurement experimental set-up

3.9 Gamma ray measurement

3.9.1 Detection

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 from occasional contaminations.

The accumulation of gamma at a particular energy generates a Gaussian curve (Reif, 1965; Syed, 2007) the area of which is proportional to the radioactivity of the characteristic radionuclide. Comparing against standards allow the establishment of relationship that can be used to determine the abundance of a particular element and elements.

3.9.2 Amount of Neutrons (Neutron flux)

Neutron flux (Krane, 1988) is the amount of neutrons available for irradiation. It is given by the expression:

$$\Phi = \frac{A\ (dn/dt)(\exp(\lambda t_d))}{KmN_A f\sigma\theta\epsilon_g\ (1-e^{-\lambda t})(1-\ e^{-\lambda t_m})} \tag{29}$$

Where: dn/dt = Activity of isotopes produced

= decay constant =0.693 / $t_{1/2}$

m= mass

N_A=Avogadro's number

f= natural abundance of isotopes

A= atomic weight

Φ= flux of thermal neutron

ϵ_g = Geometrical dependence efficiency of gamma ray of interest

θ = % intensity of gamma ray

K= self-absorption coefficient

t_d=cooling time (time after stopping irradiation & starts counting)

t= time of irradiation

t_m= time of counting the activity

3.9.3 Geometrical dependence efficiency of the detector:

The geometrical dependence efficiency of gamma ray at zero centimeters is given by the following figure.

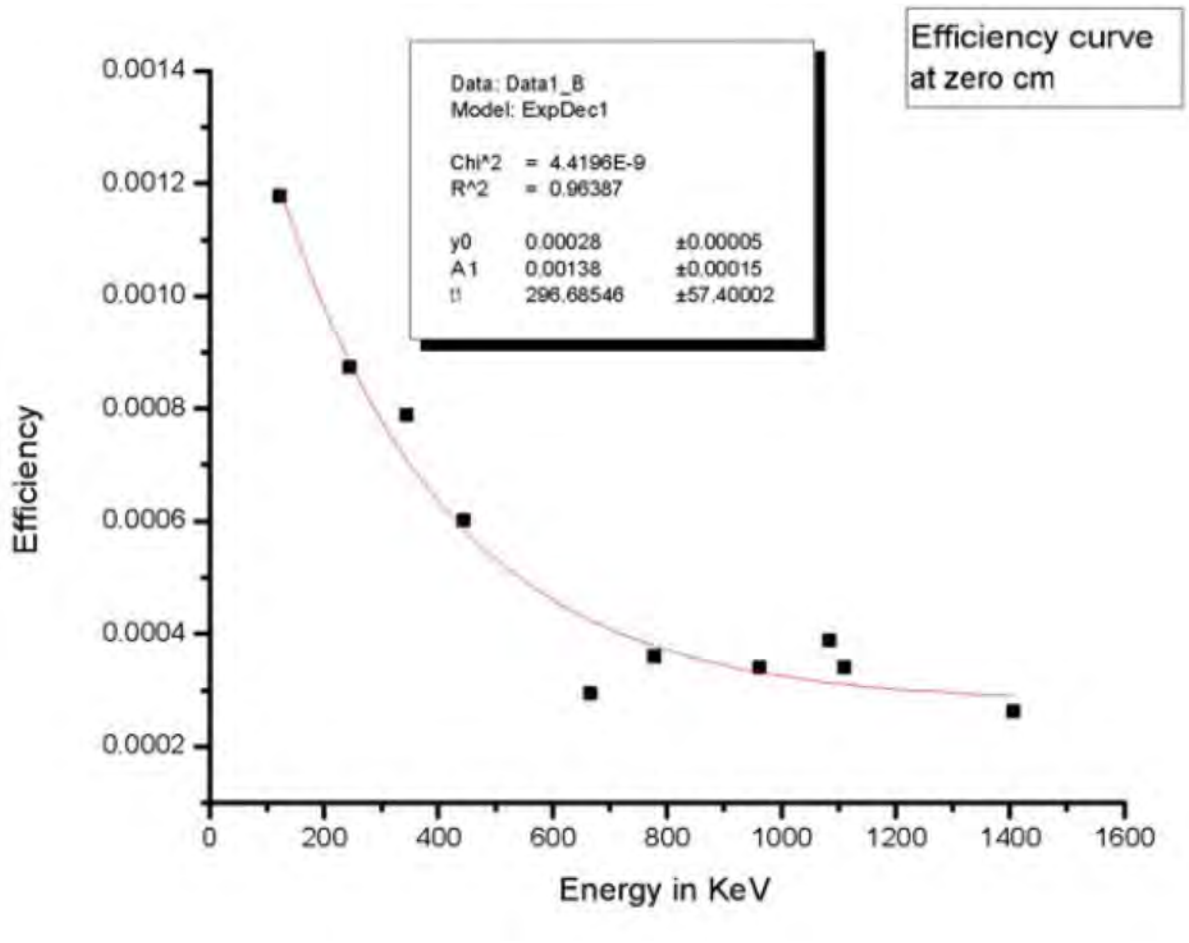


Fig (8): Geometrical dependence efficiency of the detector (Source 3)

4. Results and Discussion

4.1 Results

This thesis is conducted by keeping the beginning aim and objective. The main objective of this work was to make elemental analysis by neutron activation on the rock samples collected. Following the procedure given on the section 3.3, by setting the counting time, the gamma radiation counting job starts for the sample, iodine-1 and iodine-2 alone one by one noting the wisdrowal and transporting time. From the detected gamma radiation the following table of selected energy peak which decays through time with area covered by peak is given as:

No.	Energy (KeV)	Preset (sec)	Area	Activity (Area/Preset)	Time gap (t_{gap}) in sec	Mean time t_{mean}
1	1369.7	100	2 ± 120.78 %	0.024156	t_{g1} = 360	t_{m1} = 410
2	1369.7	100	4 ± 100.93%	0.04036	t_{g2} = 940	t_{m2} = 990
3	1369.7	200	9 ±49.72%	0.045	t_{g3} = 1514	t_{m3} = 1614
4	1369.7	500	15 ±49.39%	0.03	t_{g4} = 2470	t_{m4} = 2720
5	1369.7	1000	49 ±19.29%	0.049	t_{g5} = 4671	t_{m5} =5171
6	1369.7	1000	32 ±31.6%	0.031996	t_{g6} = 9142	t_{m6} =9642
7	1369.7	1500	45 ± 26.41%	0.03	t_{g7} = 13983	t_{m7} =14738
8	1369.7	2000	60 ±20.11%	0.029999	t_{g8} =72571	t_{m8} = 73571
9	1369.7	2000	31 ± 39.25%	0.0155	t_{g9} =77426	t_{m9} =78426
10	1369.7	2000	10±109.65%	0.005	t_{g10}= 83538	t_{m10} =84538
11	1369.7	4000	55 ± 29.98%	0.01375	t_{g11} =88100	t_{m11}=90100

Inorder to find the half of the isotope with the corresponding energy we need to draw a graph of $\ln(Activity)$ versus t_{mean} as:

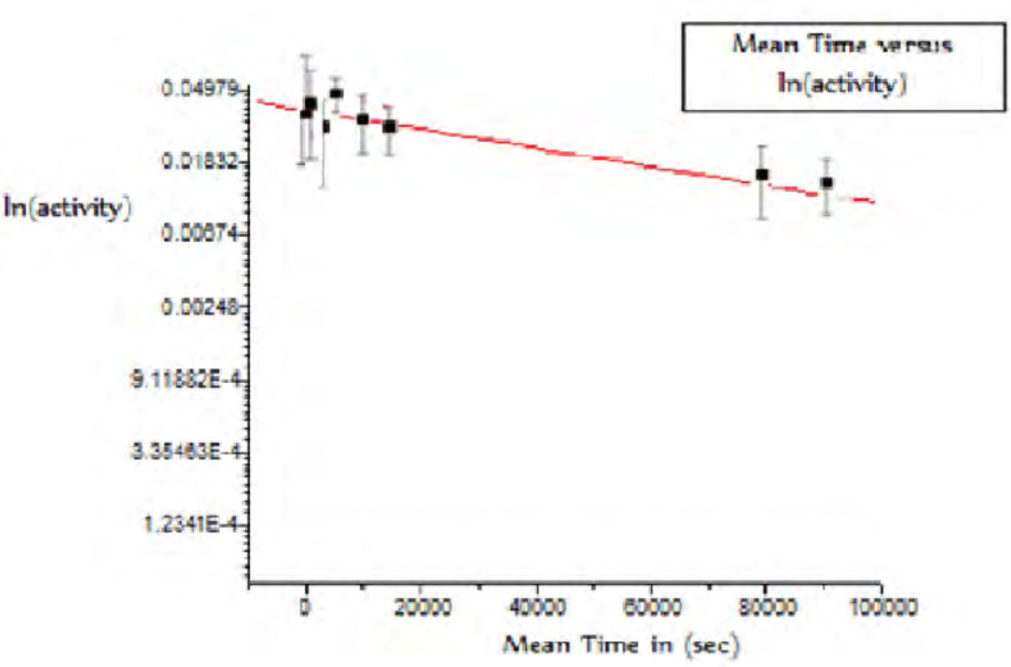


Fig 9: mean time versus ln(activity)

$$Slope = \lambda$$

$$\lambda = 1.29302 * 10^{-5}$$

$$t_{1/2} = \frac{\ln 2}{\lambda} = 53606.841\ sec \qquad (30)$$

$$t_{1/2} = 14.89079275\ hr$$

$$t_{1/2} = 14.89\ hr \qquad (31)$$

Now from isotope table having the energy 1369.7 KeV with the corresponding half-life which is 14.89 hr., it shows as this energy and half-life belongs to sodium.

Inorder to find the mass of sodium using the equation given on section 3.9.2 which is:

$$m = \frac{A\,(dn/dt)(\exp(\lambda t_d))}{K_\Phi N_A f \sigma \theta \epsilon_g\,(1-e^{-\lambda t})(1-e^{-\lambda t_m})} \qquad (32)$$

We need to have the flux (ϕ) from the data we get from the two iodines I_1 and I_2; Inorder to find the flux of our sample and for this we need the activity of both iodines, since we can have the flux of the sample by taking the mean of the two fluxes taking the samples being sandwiched by the two iodines.

Table: I1's Data recorded during radiation detection

No.	Energy (KeV)	Preset (sec)	Area	Activity (Area/Preset)	Time gap	Mean time t_{mean}
1	443.9	100	103 ± 10.86 %	1.03	t_{g1} = 530	t_{m1} = 580
2	443.9	100	111 ±10.39%	1.11	t_{g2} = 1125	t_{m2} = 1175
3	443.9	200	153 ±9.05 %	0.765	t_{g3} = 1775	t_{m3} = 1876
4	443.9	500	217± 7.68 %	0.434	t_{g4} = 2992	t_{m4} = 3240
5	443.9	1000	137 ± 12.61 %	0.137	t_{g5} = 5735	t_{m5} = 6235
6	443.9	1000	10 ± 134.56 %	0.01	t_{g6} = 10199	t_{m6} =10699
7	443.9	1500	0 ± 0.00 %	0.00	t_{g6} = 15597	t_{m6} = 16347

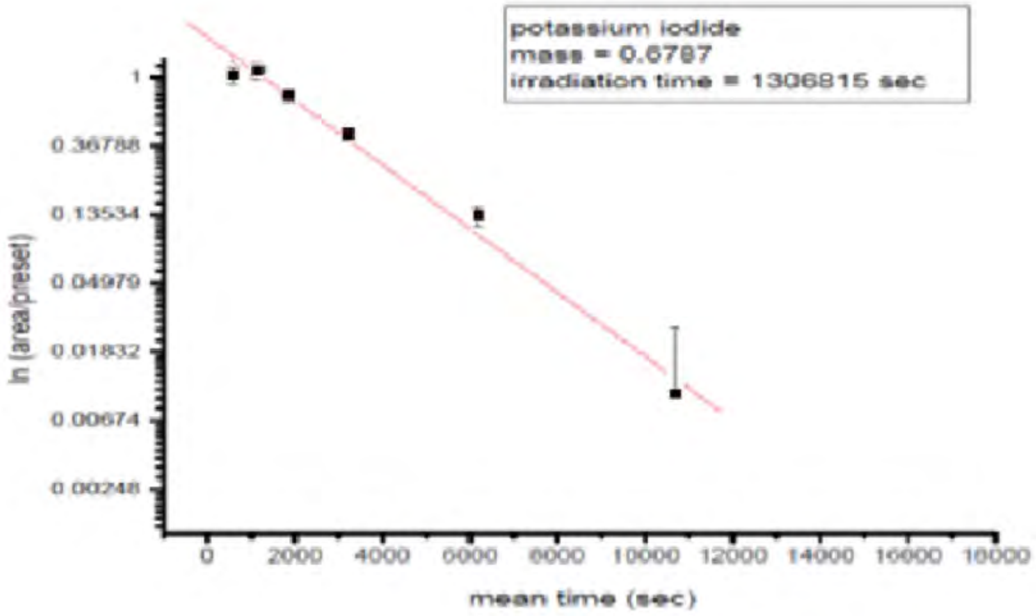


Fig 10: mean time versus ln(activity)

$$\Phi_1 = \frac{(dn/dt)(\exp(\lambda t_d))}{K\left(\frac{m_{I1} N_A f_I}{A}\right)\sigma\theta_I\epsilon_g\,(1-e^{-\lambda t})(1-e^{-\lambda t_m})} \tag{33}$$

$$\Phi_1 = 5.86613336 * 10^3 \ n/sec.cm^2$$

Table: I2’s Data recorded during radiation detection

No.	Energy (KeV)	Preset (sec)	Area	Activity (Area/Preset)	Time gap	Mean time t_{mean}
1	443.9	100	115 ± 10.18 %	1.15	t_{g1} = 688	t_{m1} = 738
2	443.9	100	87 ±11.57 %	0.87	t_{g2} = 1270	t_{m2} = 1320
3	443.9	200	147 ±8.97 %	0.735	t_{g3} = 2029	t_{m3} = 2129
4	443.9	500	162 ± 9.68 %	0.3564	t_{g4} = 3606	t_{m4} = 3856
5	443.9	1000	92 ± 18.12 %	0.092	t_{g5} = 6858	t_{m5} = 7359
6	443.9	1000	14 ± 95.56 %	0.014	t_{g6} = 11347	t_{m6} = 11847

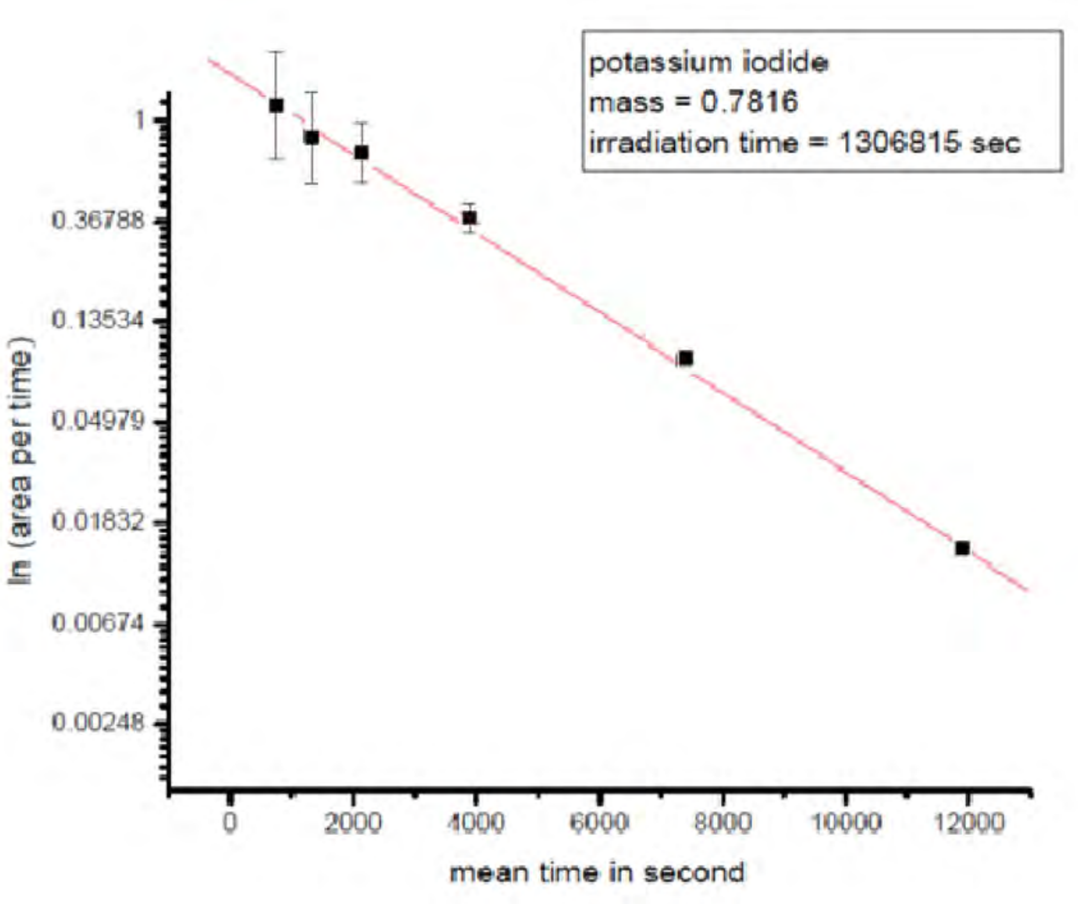


Fig 11: mean time versus ln(activity)

$$\Phi_2 = \frac{(dn/dt)(\exp(\lambda t_d))}{K\left(\frac{m_{I2} N_A f_I}{A}\right)\sigma \theta_I \epsilon_g \, (1-e^{-\lambda t})(1- e^{-\lambda t_m})} \tag{34}$$

$$\Phi_2 = 4.378746287 * 10^3 \ n/sec.cm^2$$

Now from the above two fluxes of the two iodines we can have the flux of the sample by using the following equation:

$$\phi_{sample} = \frac{\phi_1 + \phi_2}{2} \tag{35}$$

$\phi_{sample} = 5.122439824 * 10^3 \ n/sec.cm^2$

Calculating the mass of sodium:

We calculate the mass of sodium by using equation (32) since all the required are given from isotopic table and the flux is calculated above.

$$m_{Na} = \frac{A_{sample}\,(dn/dt)_{Na}(\exp(\lambda t_d))}{K \Phi_{sample} N_A f_{Na} \sigma_{Na} \theta_{Na} \epsilon_g \, (1-e^{-\lambda t})(1- e^{-\lambda t_m})} \tag{36}$$

$m_{Na} = 0.034861216 \ gm$

$m_{Na} \approx 0.035 \ gm$

The mass of sodium in 2.103 gm. of irradiated sample is 0.034861216 gm.; which means 1.65% of the irradiated sample is sodium.

4.2 Discussion

Inorder to calculate the half-life of the observed energy from the data collected by detecting radiation from the irradiated sample, the slop of the graph activity versus mean time is calculated. The mean time is calculated by noting the time at which irradiation of the sample stops, transporting time (from radiation source to the detector) and counting time as:

$$t_{mean} = time\ gap + \frac{1}{2}\ (counting\ time) \tag{37}$$

The mean time is calculated for all eleven counted trial data as:

$$\textstyle\sum_{i=1}^{11}((\,t_{mean_i} = tg_i + \frac{1}{2}\ (counting\ time)_i) \tag{38}$$

The time gap for each trial is calculated by adding the previous time gap and the previous counting time since the sample also decay during the counting process. During calculating the mean time taking the perfect micro second counting was not perfectly possible.

The activity were calculated by dividing the area with the corresponding counting time (preset) and taking the natural logarithm of the activity with the corresponding mean time the graph is drawn as given in fig (2). And from the shape the decay constant (λ) is found; by using the equation,$t_{1/2}\ {}^{\ln 2}/_{\lambda}$, the half-life is calculated as 14.89 hr. from the half-life and the energy (1369.7) the element is known to be sodium. This is not the only element in the sample but due to time constraint the only fully analyzed element is sodium.

After knowing the element, the mass of the element is calculated by using equation (32), since the sample is sandwiched between the two iodines, the flux is expected to be known from the flux of the two iodines; by taking the mean. The flux of the sample become 2.602795516 * 103 n/sec.cm2 and by using equation (32), the mass of sodium in the sample of 2.103 gm. was calculated to be ≈ 0.035 gm. which means 1.65% of the sample hold is sodium.

5. Conclusion

While preparing the sample for irradiation, great care should be taken from contamination; this is while grinding, collecting the sample and placing the powdered sample inside the sample holder. For better number of radioactivity (number of energetic radiation detection) the sample should stay inside the irradiation source as longer time as possible.

The sample which were collected from the mountains found in Woldia were irradiated being in a sample holder, having mass of 2.103 gm. and the analysis shows that the sample holds 0.035 gm. Of sodium which means 1.65% of the sample is sodium atom; this shows as the study area holds many sodium atom.

Recommendation

In order to perform this work as it was desired, there were some difficulties which has to be solved and there were also interesting and special parts which brings me to other future extended works; thus the following ideas can be recommended:

- The irradiation time of the sample should be a month or more for better population of different radioactive nuclei. Such thesis works should not be given only a semester time but may be two semester; Because some time is also involved in collecting the samples from different areas, where ever the location is, budgeting should also be considered.
- Such experimental works should be experienced by different researchers in order to have brief information about the elemental contents of different samples found here in Ethiopia.
- Out of sodium there are different elements expected in the sample from the collected data, such as Arsenic (As), Potassium (K), Copper (Cu) and many others can be found having an appropriate irradiation time.

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