

Gamma Ray Spectrometry

Practical Information

Document compiled by:

Nafaa Reguigui

September 2006.

Gamma Ray Spectrometry

1. OBJECTIVES

2. INTRODUCTION

3. INSTRUMENTATION BASICS

- 3.1. Nuclear instrumentation module (NIM)
- 3.2. Preamplifiers
- 3.3. High voltage power supply
- 3.4. Amplifier
- 3.5. Multichannel analyzer (MCA)
- 3.6. Analog to digital conversion (ADC)
- 3.7. Modular detector electronics
- 3.8. Digital signal processing (DSP)

4. THE DETECTOR

- 4.1. Germanium detectors
- 4.2. Detector efficiency
- 4.3. Detector resolution
- 4.4. Use of semi-conductor detectors
 - 4.4.1. *Detector size*
 - 4.4.2. *Photon energy:*
 - 4.4.3. *Casing material:*
- 4.5. Construction of semi-conductor detectors
- 4.6. Liquid nitrogen (LN2) cooled detectors
- 4.7. Germanium detectors with inert shields
- 4.8. NaI(Tl) scintillation detectors

5. SPECTRUM FORMATION

- 5.1. Origin of X and γ Radiation
- 5.2. Photon interaction with matter
 - 5.2.1. Photo-electric effect
 - 5.2.2. Compton effect
 - 5.2.3. Pair production
 - 5.2.4. Combined effect
- 5.3. Radiation attenuation
- 5.4. Pulse height spectrum
- 5.5. Energy transferred to the detector
- 5.6. Spectrum components
 - 5.6.1. Basic gamma spectroscopy full energy peak (FEP)
 - 5.6.2. Compton continuum and Compton edge
- 5.7. Other components of spectrum

- 5.7.1. Sum peak
- 5.7.2. Single escape peak
- 5.7.3. Double escape peak
- 5.7.4. Annihilation Peak
- 5.8. Influence of the surrounding material on the detector
 - 5.8.1. Backscatter peak
 - 5.8.2. Characteristic x-rays

6. SPECTRUM CALIBRATION AND SPECTRUM ANALYSIS

- 6.1. Spectral analysis software
- 6.2. System calibration and characterization
 - 6.2.1. Energy calibration
 - 6.2.2. Peak form and energy resolution
 - 5.2.3. Efficiency calibration
- 6.3. Analysis of the pulse height spectrum
 - 6.3.1. Peak search and deconvolution
 - 6.3.2. Finding regions of interest
 - 6.3.3. Nuclide identification
 - 6.3.4. Reliability of the nuclide identification
 - 6.3.5. Peak resolution
 - 6.3.6. Peak area determination
 - 6.3.7. Activity calculation
 - Qualitative analysis of unknown samples
 - 6.3.8. Defining a new nuclide library
 - 6.3.9. Peaks not found in the spectrum
 - 6.3.10. Background corrections
 - 6.3.11. Distortions of the pulse train due to pile-up
 - 6.3.12. Dead-time losses
 - 6.3.13. Time measurement and corrections
 - 6.3.14. Minimum detectable activity (MDA)

7. SPECIAL TOPICS

- 7.1. Low level gamma ray counting
- 7.2. High count rate gamma ray systems
- 7.3. In situ gamma spectroscopy

APPENDICES

- A. Glossary**
- B. More information**
- C. Manufacturer information:**

Gamma Ray Spectrometry

1. OBJECTIVES

This course will allow you to understand the basic theory of gamma ray spectrometry. On completion of this course, you should acquire a general knowledge about the interaction of gamma-rays with matter, and how this is applied to gamma-ray spectrometry using solid state detectors (in particular, a High-Purity Germanium Detector, HPGe). You will be able to diagram a gamma spectroscopy system and draw and explain principle components of a gamma spectrum. You should be able to apply under practical situations, the essential concepts of the technique such as spectrum analysis, energy and efficiency calibration, nuclide identification, radioactivity quantification, and different corrections applied in data reduction.

2. INTRODUCTION

Gamma ray spectrometry is an analytical method that allows the identification and quantification of gamma emitting isotopes in a variety of matrices. In one single measurement and with little sample preparation, gamma ray spectrometry allows you to detect several gamma emitting radionuclei in the sample. The measurement gives a spectrum of lines, the amplitude of which is proportional to the activity of the radionuclide and its position on the horizontal axis gives an idea on its energy.

Applications of gamma ray spectrometry include:

- monitoring in nuclear facilities,
- health physics,
- nuclear medicine,
- research in materials,
- bioscience,
- environmental science, and
- industrial uses of radioisotopes.

A conservative estimate is that over 200,000 gamma-ray spectrometers are in use in academic and industrial labs and facilities throughout the world. Because of the highly technical nature of this technique, the training of scientists and engineers in this area has been a continuing challenge.

3. INSTRUMENTATION BASICS

A typical analog HPGe detector-based gamma spectroscopy system consists of a [HPGe detector](#), [high voltage power supply](#), [preamplifier](#) (which is usually sold as part of the detector), [amplifier](#), [Analogue to Digital Converter \(ADC\)](#), and [Multi-Channel Analyzer \(MCA\)](#).

The analog system components are available in several different types, allowing the system to be tailored to the precise needs of the application and the available budget. For example, low-end amplifiers offer basic capabilities, but users with higher count rate or [resolution](#) requirements may consider amplifiers with [Pileup Rejection/Live Time Correction \(PUR/LTC\)](#) feature and both Gaussian and triangular [shaping](#). Similarly, the ADC could be either an economical [Wilkinson ADC](#) or a faster [Fixed Dead Time \(FDT\)](#). The components are usually chosen for their stability and linearity.



a) Typical NIM-based electronic setup



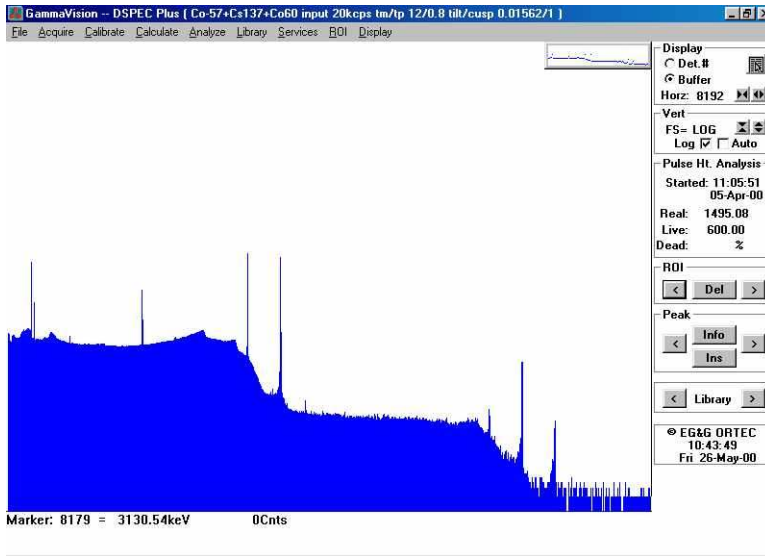
b) The rear of the setup

Figure 1. A gamma-ray spectrometer NIM-module electronic setup

Recently, [DSP](#) configurations replace the amplifier and ADC with digital signal processing electronics.

The function of the electronic system is the collection of the electrons produced from the signal pulses and the processing of those pulses and sorting them by height or energy. This process can be described by the following steps

- Photon interacts with the detector crystal, produces burst of electrons
- Applied bias voltage sweeps electrons from crystal
- Current produced by electrons forms signal pulse
- Pulse size is increased with a preamplifier
- Pulse is further intensified and shaped with amplifier
- Pulse intensity is converted into numerical value using ADC
- Numerical values are sent to MCA



A computer is required most of the times in order to visualize the spectrum and perform basic spectrum analysis using spectrum analysis software. Figure 2 shows a typical spectrum output and Figure 3. shows a block diagram for a basic gamma spectrometry system.

Figure 2. Typical Gamma ray Spectrum output

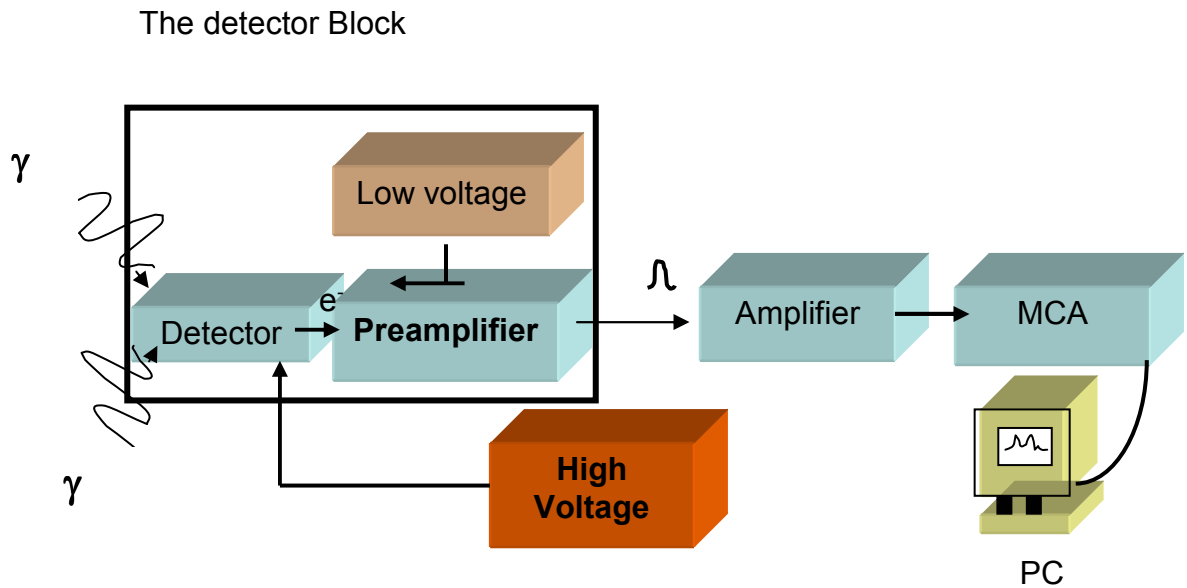


Figure 3. Block diagram of a basic gamma spectrometry system

3.1. Nuclear instrumentation module (NIM) bin – Low voltage power supply

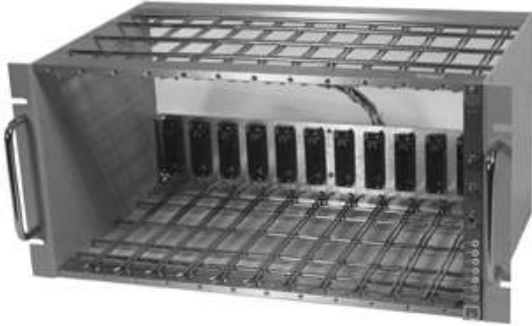


Figure 4. A typical NIM bin

The nuclear electronics industry has standardized the signal definitions, power supply voltages and physical dimensions of basic nuclear instrumentation modules using the Nuclear Instrumentation Methods (NIM) standard initiated in the 1960s. This standardization provides users with the ability to interchange modules, and the flexibility to reconfigure or expand nuclear counting systems, as their counting applications change or grow.

3.2. Preamplifiers

The charge created within the detector after the photon interaction with the detector crystal, is collected by the preamplifier. Additionally, the preamplifier also serves to provide a match between the high impedance of the detector and the low impedance of coaxial cables to the amplifier, which may be located at great distances from the preamplifier.

Most Germanium detectors in use today are equipped with [RC-feedback, charge sensitive preamplifiers](#). These can have various modes of operation: current-sensitive, voltage-sensitive and charge sensitive. [Charge-sensitive preamplifiers](#) are commonly used for most solid state detectors.

In charge-sensitive preamplifiers, an output voltage pulse is produced that is proportional to the input charge. To maximize performance, the preamplifier should be located at the detector.

When a Coaxial Germanium detector is used in applications requiring high throughput, the [Transistor Reset Preamplifier \(TRP\)](#) is favored over traditional RC feedback Preamplifiers. The higher cost of the TRP is justified by its much higher energy rate capacity, an enhancement obtained by replacing the Feedback Resistor of a typical RC feedback preamplifier with a special reset circuit.

3.3. High voltage power supply



The High Voltage Power Supply unit supplies the necessary high voltage to the detector and the necessary voltages to the rest of the system components. These units are usually able to supply up to 5000 V. Typical HPGe detectors require about 3000 V.

Figure 5. A typical HV power supply

3.4. Amplifier



The amplifier serves to shape the pulse as well as further amplify it. In this module the size (height) of the pulses are increased, and their form carefully manipulated to eliminate problems with electronic noise, shifts in the baseline, and pulses "riding" on the tails of those preceding them.

Figure 6. A typical Amplifier

3.5. Multichannel Analyzer (MCA)

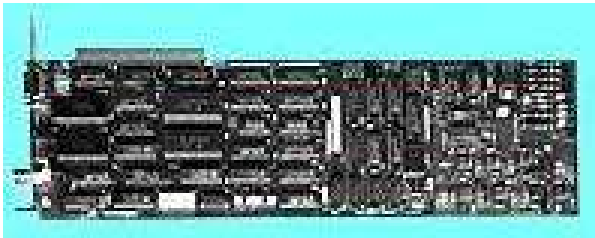


Figure 7 . A typical MCA card

The multichannel analyzer (MCA) is the heart of most experimental measurements. It performs the essential functions of collecting the data, providing a visual monitor, and producing output, either in the form of final results or data for later analysis

The multichannel analyzer (MCA) consists basically of an [analog-to-digital converter \(ADC\)](#), control logic, memory and display. The multichannel analyzer collects pulses in all voltage ranges at once and displays this information in real time.

The number in the ADC counter is volatile and must somehow be "stored" for later use. The storage process can be described by the following steps:

- The contents of the ADC counter (a number) is used as an address to identify a counter (or channel) of the **multi channel analyzer** (MCA)
- The contents of this channel is increased by "1".
- A MCA used for gamma spectroscopy contains 4096 (4K), 8192 (8 K) or 16384 (16 K) channels. If a conversion gain equivalent to 0,25 keV/channel is used with an 8 K MCA, the energy region of 0 to 2000 keV can be covered.
- Each channel has the capacity to store at least 106 pulses.

- The contents of the different channels (i.e. number of counts) as function of the channel number (i.e. address), is referred to as the **pulse height spectrum**; both for analog and digital representation.

Data acquisition modes can be of two types:

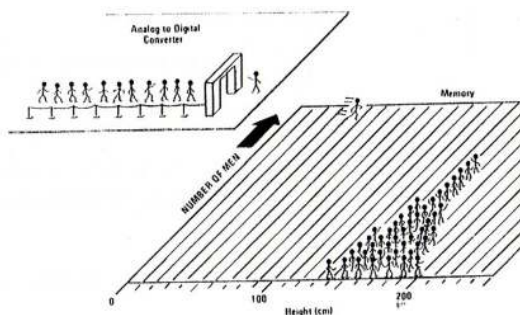
- Pulse Height Analysis (PHA)
- Multi-input PHA

In the **PHA** operating mode, a train of pulses from a radiation detector are applied to the input of the MCA. These pulses have amplitudes (heights) which are proportional to the energies of the incident radiation that was absorbed by the detector. By counting the number of occurrences of pulses of each height and forming a histogram, the MCA records the radiation energy spectrum as seen by the detector.

A better term for Pulse Height Analysis would be Pulse Height Distribution Analysis, because the input pulses are sorted by voltage amplitude to yield a histogram representing frequency of occurrence versus pulse height.

An MCA utilizing **multi-input PHA** allows simultaneous collection and storage of multiple spectra. This is typically accomplished using a Multiplexer and can be used for low count rate/low resolution counting applications. Each detector input goes to the same ADC, but a different section of memory is selected by routing bits. For low to moderate count rates, a multiplexer provides an inexpensive way to collect data from several detectors at the same time.

3.6. Analog to digital conversion (ADC)



The analog-to-digital conversion module (ADC) forms the heart of the gamma spectrometer. It converts the analog information from the pulse train into a digital format that can be stored and processed by a computer. For each analog pulse received by the ADC, a number is generated that is proportional to the height of that pulse

Figure 8: Schematic of analog to digital conversion

3.7. Modular detector electronics

In the past several years, the industry trend has been to offer modular detector electronics with the multichannel analyzer (MCA) and all supporting instrumentation for spectroscopy with a single detector combined in a compact,

stand-alone enclosure. These modular MCAs are smaller, lighter and use less power than the NIM-based counting systems that preceded them. However, their performance is equal to, or greater than, comparable NIM-based systems.

3.8. Digital signal processing (DSP)



High performance gamma spectrometers are often designed today using Digital Signal Processing (DSP) techniques rather than analog shaping amplifiers. The shaping functions are then performed in the digital domain rather than with analog circuitry. DSP filters and processes the signals using high speed digital calculations rather than manipulation of the time varying voltage signals in the analog domain.

Figure 9: DSP module (ORTEC)

4. THE DETECTOR



The detector is the center piece of the gamma spectroscopy system. The gamma photons interact with the detection material and transfer their energies to electrons or to positrons in the case of annihilation. These produced particles lose their energy within the detector, creating ionized atoms and ion pairs. These secondary entities form the basis of the detector signal. High purity Germanium is the mostly used material for gamma ray spectrometry systems.

For the past 20 years, Ge detector performance has been specified using the methods and values specified in “IEEE Standard Test Procedures for Germanium Gamma-Ray Detectors”, ANSI/IEEE Std 325-1986

.Figure 10: Different detectors

These specifications are the FWHM, FW.1M, FW.02M, Peak-to-Compton ratio, and relative efficiency. These standard specifications are useful in guiding the user to an appropriate detector choice for the intended measurement.

4.1. Germanium detectors

Germanium detectors are semiconductor diodes having a P-I-N structure in which the Intrinsic (I) region is sensitive to ionizing radiation, particularly X-rays and gamma rays. Under reverse bias, an electric field extends across the intrinsic or depleted region. When photons interact with the material within the depleted volume of a detector, charge carriers (holes and electrons) are produced and are swept by the electric field to the P and N electrodes. This charge, which is in proportion to the energy deposited in the detector by the incoming photon, is converted into a voltage pulse by an integral charge sensitive preamplifier.

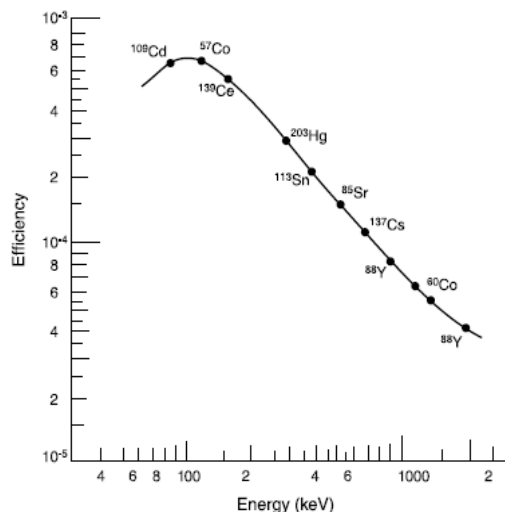
4.2. Detector efficiency

Generally, the sensitivity of a HPGe system will be in direct proportion to the detector efficiency. The efficiency of a detector is a measure of how many pulses occur for a given number of gamma rays, i.e., the ***fraction*** of all the photons that are emitted by the source or sample (***A***), which cause an event in the detector (***N***). This is the **efficiency** ε of that detector.

$$N = \varepsilon \cdot A$$

Various kinds of efficiency definitions are in common use for gamma ray detectors. There is a lot of confusion surrounding this concept because users are often careless about what is meant by "an event in the detector". For some it means "a full energy event", and for others it means "any event that can be measured".

- a. **Absolute Efficiency:** The ratio of the number of counts produced by the detector to the number of gamma rays emitted by the source (in all directions).
- b. **Intrinsic Efficiency:** The ratio of the number of pulses produced by the detector to the number of gamma rays striking the detector. The **intrinsic** efficiency of a detector is defined in terms of the number of photons in a collimated beam incident on its entrance window; and not by the number of photons emitted by the source. This definition has the advantage that it is not tied to any source-detector geometry. It is often used to compare different detectors; but is of little importance when measuring the activity of radioactive samples.
- c. **Relative Efficiency:** HPGe detectors are almost universally specified in terms of their relative full-energy peak efficiency compared to that of a 3 in. x 3 in. NaI(Tl) Scintillation detector at a detector to source distance of 25 cm at 1.33 MeV..
- d. **Full-Energy Peak (or Photopeak) Efficiency:** The efficiency for producing full-energy peak pulses only, rather than a pulse Efficiency curve of any size for the gamma ray. It is clear from the previous discussion that the photopeak efficiency of a detector will be dependent on the energy of the photon interacting with it



Clearly, to be useful, the detector must be capable of absorbing a large fraction of the gamma ray energy. This is accomplished by using a detector of suitable size, or by choosing a detector material of suitable high Z. Detectors of greater than 100% relative efficiency have been fabricated from germanium crystals ranging up to about 75 mm in diameter. About two kg of germanium is required for such a detector. An example of a full-energy peak efficiency curve for a germanium detector is shown in Figure 11

Figure 11 Full-energy peak efficiency curve for a germanium detector

4.3. Detector resolution

Resolution is a measure of the width (**full width half max, FWHM**) of a single energy peak at a specific energy, either expressed in absolute keV (as with **Germanium Detectors**), or as a percentage of the energy at that point (**Sodium Iodide Detectors**). Better (lower **FWHM** value) resolution enables the system to more clearly separate the peaks within a spectrum. Figure 12 shows two spectra collected from the same source, one using a sodium iodide (NaI(Tl)) detector and one using germanium (HPGe). Even though this is a rather simple spectrum, the peaks presented by the sodium iodide detector are overlapping to some degree, while those from the germanium detector are clearly separated. In a complex spectrum, with peaks numbering in the hundreds, the use of a germanium detector becomes mandatory for analysis.

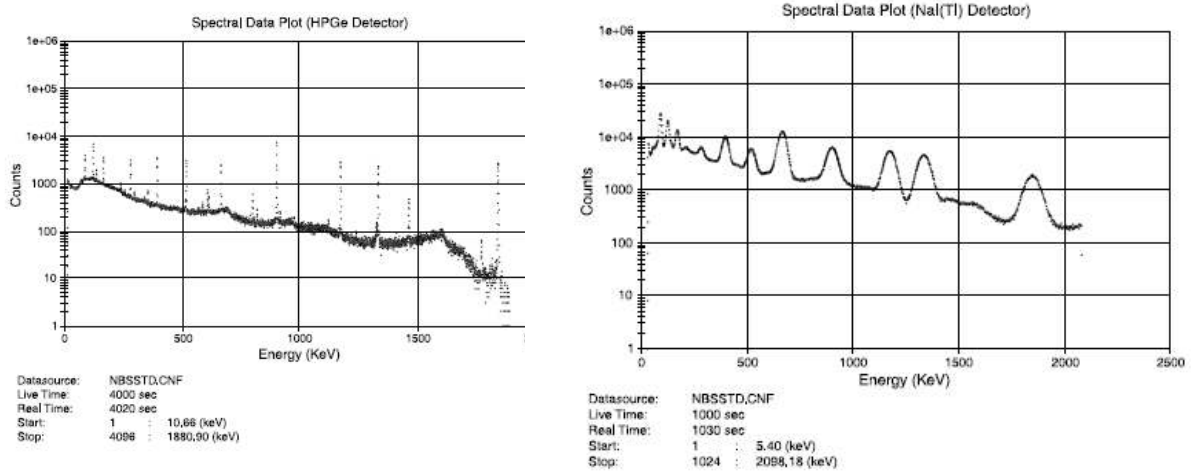


Figure Comparison between HPGe and NaI detectors

4.4. Use of semi-conductor detectors

The counting efficiency of HPGe detectors is controlled by three major considerations:

- *Detector size*
- *Photon energy*
- *Casing material*

4.4.1. Detector size

The larger the volume of the crystal, the larger the possibility that a photon will transfer all its energy to the Ge, and the higher the overall efficiency in general. *(The prices of these detectors also rise dramatically with increase in their volumes !)*



a) An 80 % HPGe

b) A 35 % HPGe

Figure 13 Two HPGe Coaxial detectors of different sizes (relative efficiencies)

4.4.2. Photon energy:

The efficiency value decreases for high energy photons (typically above 300 keV); approximately as $\varepsilon \propto E_{\gamma}^{-0.5}$. At lower values for the photon energy the photoelectric effect becomes very strong, and every photon falling in on the detector face is completely absorbed. When this state of 100 % absorption of photons is reached (typically below about 100 keV), the intrinsic efficiency of the detector stays constant for the soft γ -photons (*it can not increase to more than 100 %*).

4.4.3. Casing material:

The detector is kept inside a small vacuum container to keep it at a low temperature. The canning is normally made from stainless steel to obtain a rigid container. This material will absorb some of the photons falling in on the entrance window before they can reach the Ge-material. This absorption in the window increases rapidly for energies below 100 keV. A combination of the last two effects results in a drop in overall efficiency at lower energies, and Ge-detectors can normally not be used below 80 keV.

The low energy range of the Ge-detector can be extended to about 50 keV by using aluminum as canning material, or even to about 30 keV using a beryllium entrance window. The semi-conducting material used for these low-energy detectors is usually more flat (i.e. a pancake form), than those used in high-energy work (similar dimensions for thickness and diameter). It is therefore important to place the sample to be counted in front of the entrance window.

One can obtain special detectors with a hole drilled into the detector material (and the canning too). These "well type" detectors have the advantage that a small sample can be placed inside the well, thereby maximizing the absolute efficiency of the detector. The diameter of the hole is normally less than 20 mm,

and the detector can only be used for small samples. If sufficient sample material is available, a large sample outside the detector would probably give a higher count rate than a very small amount inside the well.

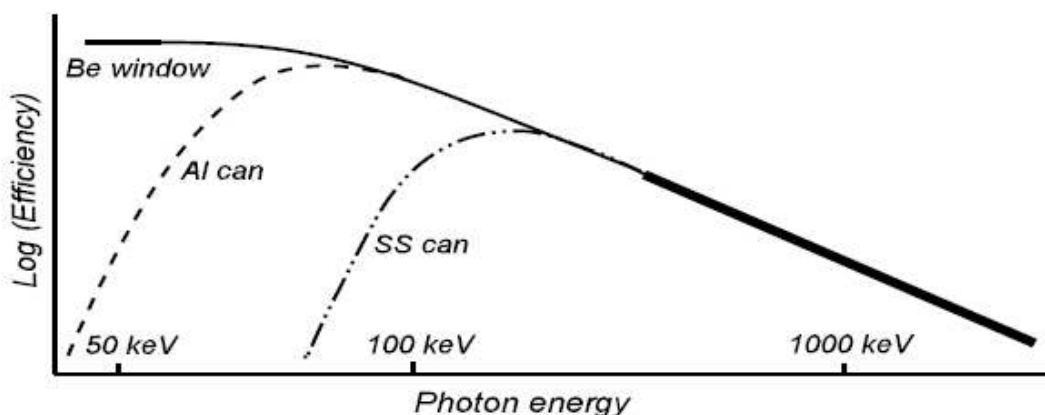


Figure 14 Effect of photon energy on the counting efficiency of a HPGe detector

4.5. Construction of semi-conductor detectors

The detector suitable for high-energy γ -spectrometry is normally a cylinder of high-purity germanium with a diameter of about 50 mm and height of 30 mm. Larger blocks are also available, but the price increases rapidly with the size. The front face and circumference of the block are plated with a metal layer which acts as the one electrode (connected to earth). A small hole is drilled in the reverse side of the block; into which the other electrode is inserted. This electrode, which is spring-loaded to ensure proper contact, is kept at a high positive bias to collect all the free electrons produced during the interaction of the γ -photon.

The detector material rests on the one end of a thick copper rod. The other end of the rod is kept in liquid nitrogen (at - 196 C) to cool it. Copper is an excellent heat conductor, and this arrangement ensures that the detector itself is always cooled. The block, some of the electronic components and part of the copper rod are enclosed in a small vacuum container. No heat is conducted in vacuum, and the outer surface of this chamber will be at room temperature even when the detector inside it is at - 196 C. This prevents water vapor from the air condensing on the detector.

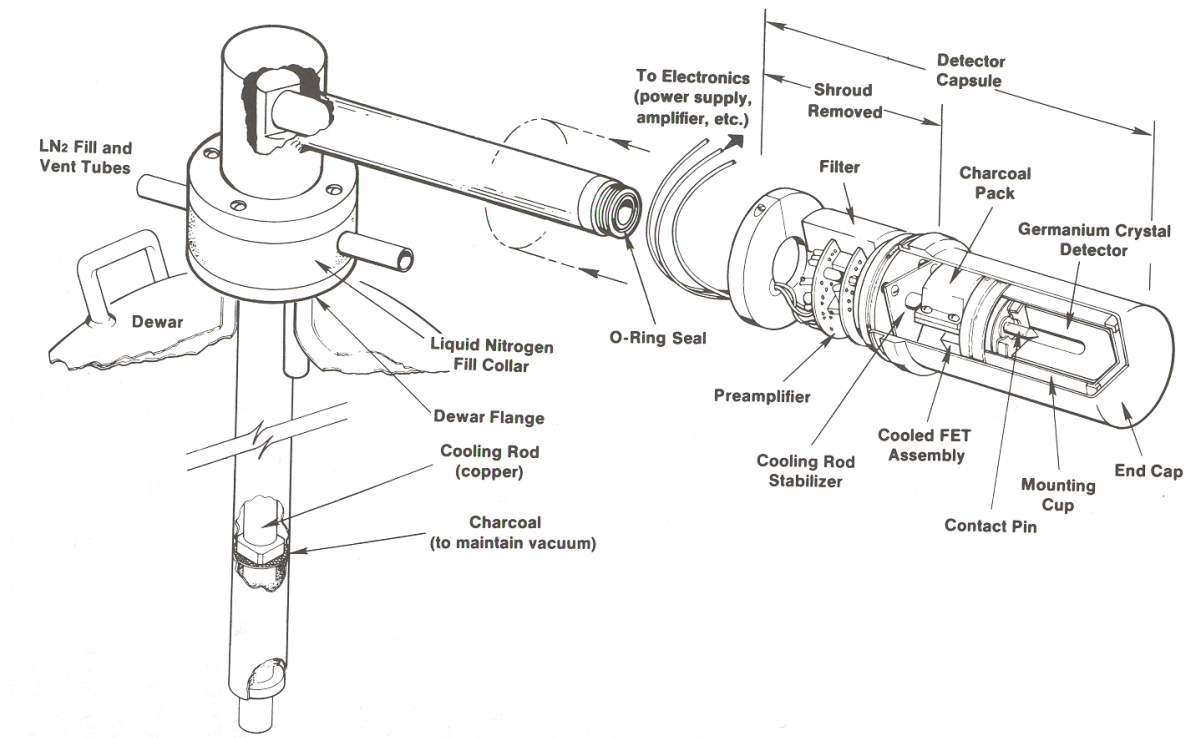


Figure 15 HPGe Detector Operational Characteristics - Cryostat and Dewar

4.6. Liquid nitrogen (LN2) cooled detectors

Any HPGe detector requires a supply of liquid nitrogen to keep it at a very low temperature. Because germanium has a relatively low band gap, these detectors must be cooled in order to reduce the thermal generation of charge carriers (thus reverse leakage current) to an acceptable level. Otherwise, leakage current induced noise destroys the energy resolution of the detector. Liquid nitrogen, which has a temperature of 77°K or -196 °C is the common cooling medium for such detectors. The detector is mounted in a vacuum chamber which is attached to or inserted into an LN2 Dewar or an electrically powered cooler. The sensitive detector surfaces are thus protected from moisture and condensable contaminants. The liquid nitrogen is kept in a large cryostat that can contain sufficient quantity of the liquid to last at least a week between fillings (*NOTE: A detector face that is cold and wet is a sure sign that something is wrong with the vacuum system.*).

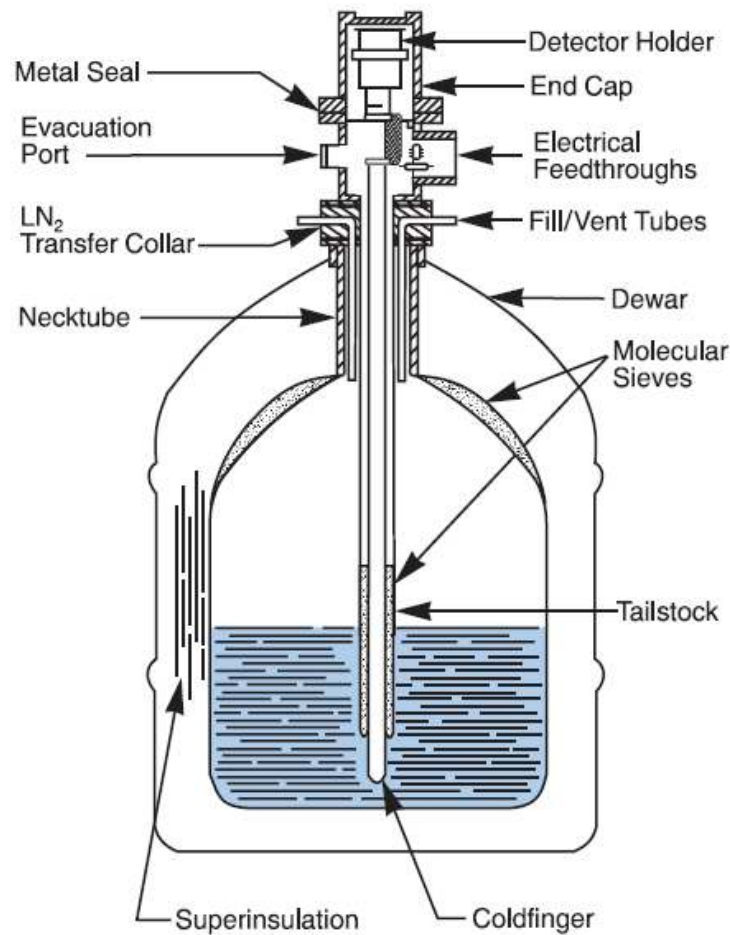


Figure 16 Model 7500SL Vertical Dipstick Cryostat (Canberra)

In electrically refrigerated detectors, both closed-cycle mixed refrigerant and helium refrigeration systems have been developed to eliminate the need for liquid nitrogen. Besides the obvious advantage of being able to operate where liquid nitrogen is unavailable or supply is uncertain, refrigerated detectors are ideal for applications requiring long-term unattended operation, or applications such as undersea operation, where it is impractical to vent LN₂ gas from a conventional cryostat to its surroundings.

4.7. Germanium detectors with inert shields

There are many different types of shield designs that are available, and because of the difficulty in determining the background contribution of the materials used in a given shield, it is difficult to assign performance levels to various types of shields. However, some criteria for shield designs have evolved over the years, such as:

1. The shield should not be designed to contain unnecessary components like the Dewar. It will only contribute to increased background if it is within the walls of the shield, as well as unnecessarily increase the shield's size, weight and cost.
2. The detector should be readily installed and removable from the shield. A HPGe detector and shield system should have a liquid nitrogen transfer system to avoid removing the detector for the weekly filling.
3. Sample entry should be convenient to the operator.
4. The shield should accommodate a variety of sample sizes and configurations. The HPGe detector should be located in the center of the shield so as to minimize scatter from the walls. In this position, the shield must accommodate the largest sample that is anticipated. Also, sample placement should be accurately repeatable and easily verified by the operator.

The performance of the shield using a CANBERRA HPGe detector is given in table 1.

Shield Specs:	Inside Dimensions Wall Thickness Material	28 cm dia. x 40.5 cm high 10 cm Low Background Lead
HPGe Specs:	Relative Efficiency Resolution	12% 1.95 keV FWHM at 1.33 MeV 0.90 keV FWHM at 1.22 keV
Background Count:	2.25 counts/second in the 50 keV–2.7 MeV range	
Sensitivity:	Table 1.4 lists the sensitivities of several single radioisotopes, assuming a counting time of 50 000 seconds, a 50% error and a detector-to-point-source distance of 1 cm.	

Table 1.4 Radioisotope vs. Sensitivity		
RADIONUCLIDE	ENERGY in keV	SENSITIVITY in pC
⁵⁷ Co	122	2
¹³⁹ Ce	165	3
¹³⁷ Cs	662	6
⁶⁰ Co	133	10

Table 1. The performance of the shield using a CANBERRA HPGe detector

The design or configuration of the cryostat is another factor in system performance. Some cryostat/shield designs do not prevent streaming from the outside environment, nor do they provide self-shielding from their own relatively

hot components. Through an improper choice of material types and/or thicknesses, the cryostat may appreciably contribute to the background.

A typical gamma spectrometry laboratory is shown in figure ...



Figure 17 A typical Gamma Spectrometry Laboratory

4.8. NaI(Tl) scintillation detectors

A gamma ray interacting with a scintillator produces a pulse of light, which is converted to an electric pulse by a photomultiplier tube. The photomultiplier consists of a photocathode, a focusing electrode and 10 or more dynodes that multiply the number of electrons striking them several times each.

The properties of scintillation material required for good detectors are transparency, availability in large size, and large light output proportional to gamma ray energy. Relatively few materials have good properties for detectors. Thallium activated NaI and CsI crystals are commonly used.

The high Z of iodine in NaI gives good efficiency for gamma ray detection. A small amount of Tl is added in order to activate the crystal, so that the designation is usually NaI(Tl) for the crystal. The best resolution achievable ranges from 7.5%-8.5% for the 662 keV gamma ray from ^{137}Cs for 3 in. diameter by 3 in. long crystal, and is slightly worse for smaller and larger sizes. Many configurations of NaI detectors are commercially available, ranging from crystals

for X-ray measurements in which the detector is relatively thin (to optimize resolution at the expense of efficiency at higher energies), to large crystals with multiple phototubes. Crystals built with a well to allow nearly spherical 4π geometry counting of weak samples are also a widely-used configuration.

Nal is still the dominant material for gamma detection because it provides good gamma ray resolution and is economical.

5. SPECTRUM FORMATION

The characteristic gamma rays of a given radionuclide that is present in the analyzed sample are emitted with one or more well known energies. These gamma rays are detected by the detector, treated by the electronic setup and then observed in the form of a spectrum. In this chapter, we will describe the different processes that are involved in the spectrum formation and study the different spectrum components.

5.1. Origin of X and γ Radiation

After a nucleus has emitted a beta particle, more often than not it is still slightly unstable and will get rid of its excess energy by emitting a gamma ray immediately afterwards. The time delay between the two emissions is so short (less than a billionth of a second) that we look upon them as being simultaneous, and as being only one disintegration.

Light, radio waves, X-rays and gamma rays are all "electromagnetic" radiation. They differ from one another only in their frequencies.

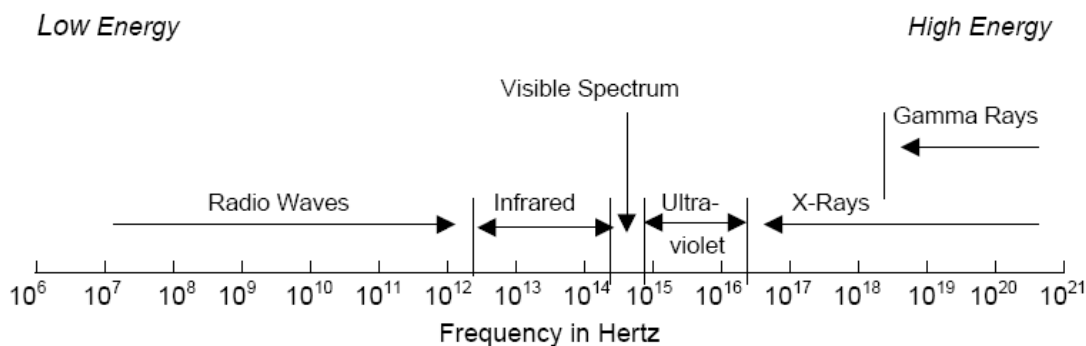


Figure 18 The electromagnetic spectrum

The energy of any electromagnetic radiation depends on its frequency. In fact, the energy and frequency are directly proportional. X and gamma rays have the highest energies. They cover about the same range of frequencies and, in fact, the only difference between them is their origin. Gamma rays are emitted from the nuclei of radioactive atoms, while X-rays are produced by disturbances in the electron orbits of atoms. X-rays are not emitted from nuclei.

5.2. Photon interaction with matter

The most important interaction of α , β and γ -rays is by **ionization**, where some electrons are ejected from neutral atoms leaving positively charged ions behind. This process can be readily visualized for the α - and β -particles which are both

electrically charged, but it is more difficult for γ -rays which are electrically neutral electromagnetic waves. It is, however, essential that the interaction be understood in order to apply the knowledge to γ -spectrometry. Gamma photons produce ionization indirectly as a result of collisions. There are three ways in which a gamma photon can interact with an atom.

- The photo-electric effect
- The Compton scattering, and
- The pair production

5.5.1. Photo-electric effect

In the photoelectric process, all the energy of the incident photon ($h\nu$) is transferred to a bound electron near the nucleus of the atom. This electron is then ejected from that atom with a kinetic energy of

$$E = h\nu - I$$

where I denotes the energy required to remove a bound electron from that atom. In this effect it is the **binding energy** for the K- and L-shell electrons to that atom.

The high energy electron is in all respects the same as a β -particle, and can cause secondary ionization in the medium. It is assumed that this electron will lose all its energy in the material. It is important to note that the photon "disappears" in this process.

The following aspects concerning the electrons are important for understanding this effect:

- The photoelectric effect involves mainly those electrons that are closely bound to the nucleus (i.e. the inner orbitals) in the K- and L-shells. The probability for the effect becomes less for the electrons that are more loosely bound in outer orbitals.
- If the energy of the incident photon is less than the ionization potential or binding energy of an electron in a particular inner shell (i.e. if $h\nu < I_K$), that electron can not be involved in the photoelectric effect. Less tightly bound electrons in other (outer) orbitals are, however, then available if $h\nu > I_L$.

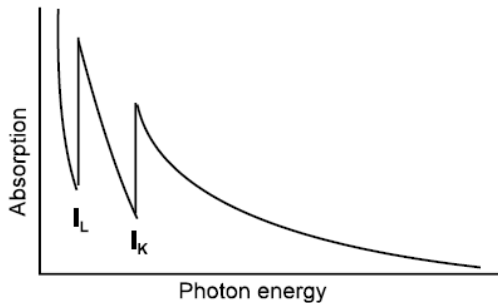
The probability for the photoelectric effect is also determined by a number of other aspects:

- The higher the energy of the incident photon (above the $h\nu > I_{\text{limit}}$), the smaller the chance for the effect to occur.

- The higher the atomic number of the absorbing material, the bigger the chance for the effect. This can be summarized by the following equation above the low-energy absorption edges:

$$\text{Probability of the effect} = k \cdot \frac{Z^5}{E^{3,5}}$$

where: **Z** is the atomic number of the absorbing material; and **E** is the energy of the photon.



The probability of the photoelectric effect as a function of the photon energy, is illustrated in the adjacent figure. Note the sharp decrease in absorption as the energy of the photon drops below the ionization potential of the K- and L-shell electrons. High energy photons absorbed by K-shell electrons, will cause the emission of K X-rays of the absorber when the vacancy in the K-shell is filled.

Figure 19 The probability of the photoelectric effect

*This principle is used in the construction of **lead shields** for gamma spectrometers. Photons from the sample and background radiation are absorbed in this shield. If it happens via the photoelectric effect, the X-rays of lead will be emitted; and measured by the detector. A 1 mm layer of a heavy metal (eg cadmium) is therefore placed inside the lead to absorb the Pb X-rays. And a 1 mm layer of copper is added on the inside to absorb the Cd X-rays.*

5.2.2. Compton effect

If the gamma photon energy is not low enough for this, **Compton scattering** is more likely. Here, the photon will transfer only part of its energy to an electron. The remaining energy is taken away by a new gamma photon of lower energy. We say that this new photon is *scattered*, because it will take off in a new direction. The electron that was ejected from the atom produces ionizations in just the same way as a beta particle.

This process differs from the photoelectric effect in that:

- (i) the photon transfers only a **fraction** of its energy to the electron, and
- (ii) only the (essentially) free electrons in the outer orbitals are involved in the process.

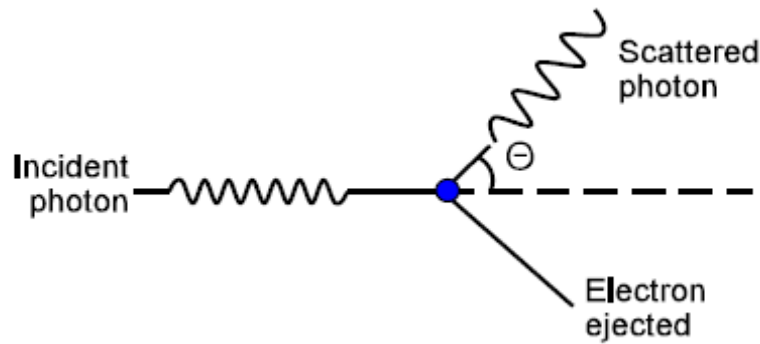


Figure 20 Compton effect

The photon is reflected (or scattered) through an angle (θ) with the respect to the line of incidence (figure 20), and moves along with decreased energy. If the photon with energy E is treated as a wave with wavelength λ , Compton scattering can be described by:

$$(\lambda - \lambda_0) = \frac{h}{mc} \cdot (1 - \cos \theta) \quad \text{where: } E_\gamma = h\nu = \frac{hc}{\lambda}$$

If the scattering angle θ is known, it is then possible to calculate from standard mechanics: (i) the angle at which the electron is moving, and (ii) the ratio in which the available energy (from the incident photon) is divided between the electron and the scattered photon.

The maximum energy that a photon can transfer to an electron is when $\theta = 180^\circ$, i.e. when the photon is scattered directly backwards (and the electron moves in the direction of the incident photon). This is still less than the total energy that can be transferred by the same photon in a photoelectric interaction. The scattering angle can be any value between 0 and 180° . And the smaller the angle, the smaller the amount of energy that is transferred to the electron. A photon just "grazing" an electron ($\theta = 0$) transfers no energy.

The probability for Compton interaction increases with the atomic number of the absorber, and decreases with the energy of the incident photon. This can be summarized by:

$$\text{Probability of the effect} = k \cdot \frac{Z}{E}$$

An interesting situation develops for the geometric arrangements normally used for counting samples: the sample is placed close to the detector inside a heavy shield (normally lead). The detector therefore "sees": (i) photons directly from the sample, and (ii) a smaller number that were all reflected by approximately 180° from the absorber. This **backscattering** produces photons with more or less the same energy, regardless of the energy of the photons incident on the lead shield. The **backscattered photons** appear as a broad peak in the low-energy region of the pulse height spectrum.

5.23. Pair production

A high-energy gamma photon sometimes changes into two electrons, one positive and the other negative. The positive electron is called the **positron**. This interaction is known as **pair production**, for obvious reasons.

This effect can be explained in terms of the relativity theory (Albert Einstein) which postulates that "matter" and "energy" are different forms of the same entity. A particle of mass ***m*** can be converted into an amount of energy ***E***, and *vice versa*, according to the equation:

$$E = mc^2$$

where *c* is the speed of light.

Pair production is one consequence of this theory: A photon with energy ***E*** is converted into two different particles with the same mass, but with opposing electrical charges. The excess energy (i.e. more than the minimum required to produce the particles) is shared by the two particles in the form of kinetic energy:

$$E_{\gamma} = (m_{+} + m_{-}).c^2 + (\frac{1}{2}mv^2)_{+} + (\frac{1}{2}mv^2)_{-}$$

The two particles involved are both electrons. One, the electron, is negatively charged, the other, is the positive electron, or **positron**. It has the same mass and nuclear properties as the negative electron, but with the opposite electrical charge. The term negatron is sometimes used to distinguish the negative from the positive electron.

The amount of energy equivalent to the mass of the electron or positron ($= 9,11 \times 10^{-28}$ g) can be calculated from the Einstein equation. It amounts to 511 keV for one particle; or 1022 keV for a positron-negatron pair. The equation above can therefore be written as:

$$E_{\gamma} = 1022 + (\frac{1}{2}mv^2)_{+} + (\frac{1}{2}mv^2)_{-}$$

All energy values are in keV.

The positron at rest is not a stable particle. Once it has been produced by pair production, the positron will lose energy by interaction with electrons in the absorbing material in the same manner that a negatron does. But when it has lost all its kinetic energy, it can not exist in the presence of other electrons, and a nuclear reaction occurs. In this reaction a positron and negatron pair destroys (annihilates) each other with the production of two photons with energy of 511 keV each. (*This annihilation can occur between the positron and **any** electron; not necessarily with the one formed during the pair production.*)

There are two photons involved in order to conserve momentum during the reaction. It can be represented by:

$$\beta^+ + \beta^- \rightarrow \gamma_1 + \gamma_2 \quad \text{and} \quad E_{\gamma 1} = E_{\gamma 2} = 511 \text{ keV}$$

Pair production is only possible if the energy of the incident photon is sufficient to provide at least the mass of an positron and negatron pair ($> 1022 \text{ keV}$). In practice it is only significant for photon energies above 1,5 MeV. The higher the photon energy, the more likely pair production is to occur. The likelihood varies roughly as $(E - 1.02)^2 Z^2$.

5.2.4. Combined effect

The three effects through which γ -rays can interact directly with, and transfer energy to matter, were discussed separately in the preceding sections. It is, however, also possible that two or more different events can occur in sequence for the same primary photon. A Compton scattered photon can be totally absorbed by a photoelectric event, or it can be scattered again by a second Compton interaction. Similarly, one or both of the photons that result from the annihilation of the negatron produced in pair production, can be scattered once or repeatedly. Two situations should be distinguished when considering the overall effect of the interaction of gamma rays with matter:

- One can consider a collimated beam of photons, and focus on what happens to this beam when it traverses a layer of absorbing material. This is normally the approach when one studies shielding and safety.
- One can also focus on the absorbing material, and try to determine how much energy was transferred to it. Since the purpose of γ -spectrometry is to determine the energy of individual photons, and the "material" is normally the detector, this will be the major concern here.

5.3. Radiation attenuation

Suppose a collimated beam of γ -rays with intensity I (photons.m²) falls on a thin layer of material that is Δx (m) thick. Should any of these particles interact with

the absorber by one of the three effects discussed above, it will disappear from the beam. This fraction, ΔI , that is removed from the beam is proportional to $I\Delta x$. Integrating this over a thick layer leads to the following attenuation equation:

$$I_x = I_0 \cdot e^{-\mu x}$$

where:

- I intensity of the beam incident on the absorbing layer;
- I_x intensity of that beam after traversing the layer;
- x total thickness of the absorbing material; and
- μ factor describing the absorption properties of the material involved, referred to as the **absorption or attenuation coefficient** of that material.

We can also write the same equation in terms of the number of incident photons N_0 and the number of the transmitted photons N :

$$N = N_0 e^{-\mu_L x}$$

Where μ_L is the linear attenuation coefficient (in m^{-1}). This coefficient is the sum of the three linear attenuation coefficients t , σ et K , corresponding to the photoelectric, Compton and pair production effects, respectively.

$$\mu_L = t + K + \sigma$$

The attenuation equation can also be written using the mass attenuation coefficient:

$$(\mu / \rho) N = N_0 e^{-\frac{\mu_L}{\rho} \rho x}$$

where ρ is the material density and $\rho x = m/s$ is the surface mass ($g \cdot m^{-2}$).

5.4. Pulse height spectrum

For gamma ray spectrometry, only the height of the pulses are important; and not the time when the events occurred. The objective is therefore to convert the train of pulses produced by the detector, into information that can be used by the analyst to determine the energy and intensity of the photons emitted by the source.

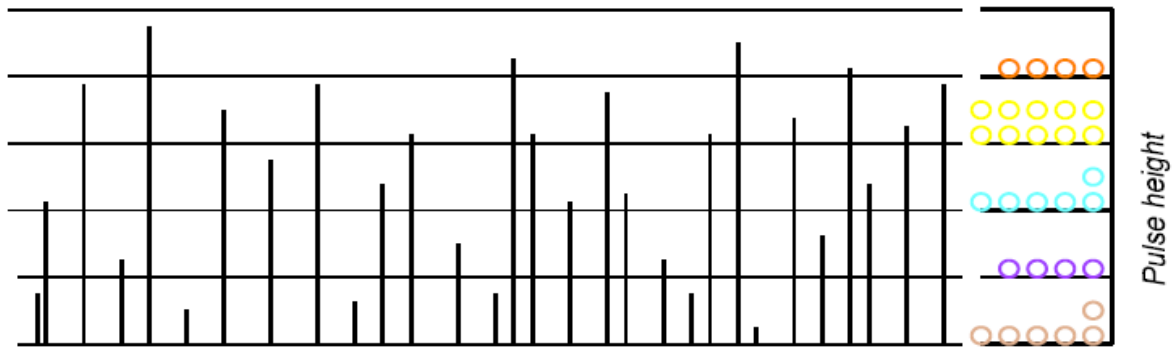


Figure 21: Train of pulses delivered at the output detector

The pulses delivered at the output of the detector are "sorted" or classified according their height as shown in figure 21. The number of pulses in each category are then counted; with each counter representing a pulse height interval. There are two concepts involved in this process:

- the index number or code of each counter. *(These codes normally run from "1" to higher values as required. They are referred to as **channels** in gamma spectrometry.)*
- the number of pulses collected in each counter (i.e. the contents of that counter).

If the **contents** of each channel is plotted (on the vertical axis) against the **number** of that channel (on horizontal axis), one obtains a **pulse height spectrum** as shown in this figure 22.

The second illustration is derived from the first one. It is, however, impossible to re-construct the original train of pulses from this figure because both their actual sizes and the time when they occurred, have been lost in the processing of the data.

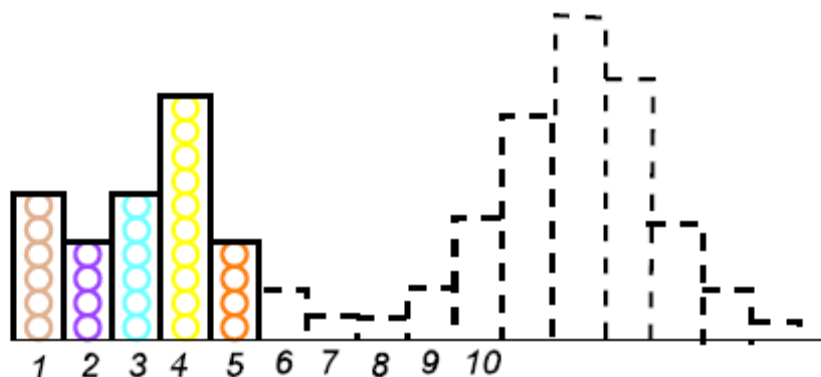


Figure 22 Pulse height spectrum

All the information contained in the first figure, can only be presented in the form of a drawing. This is referred to as information in **analog** format. The processed data, on the

other hand, are available as "numbers"; i.e. in a **digital** format. It is possible to present the digital information in analog format (e.g. when plotting digital information from a table in a graph). It is, however, necessary to realise that the analog representation of digital data in a pulse height spectrum, is different from the analog pulse train from which the digital data were derived. Computer systems can only handle digital information, and all analog data must first be converted into digital format.

5.5. Energy transferred to the detector

The prime concern for γ -spectrometry is the energy of the photon that must be measured. It is clear that the amount of energy deposited in the absorber material (i.e. the detector) will be equal to the photon energy in the following cases only (see figure 23):

- a photoelectric event;
- one or more Compton scatterings followed by a photoelectric event;
- pair production followed by photoelectric absorption of **both** annihilation photons; which might have been preceded by one or more Compton scatterings.

In each of these cases the sequence stops in a photoelectric event. It is, however, more correct to refer to these as **full energy** events, and not as "photoelectric" events.

An interesting situation can develop when a high energy photon ($E > 1022 \text{ keV}$) starts the sequence: Some of the annihilation photons can escape from the detector. No distinction can be made between the two photons produced on annihilation of the positron, and the following cases can be distinguished:

- both annihilation photons are totally absorbed: total energy event;
- only one annihilation photon is totally absorbed, while the other escapes from the detector: **single escape** event; and
- none of the two photons are totally absorbed: **double escape** event.

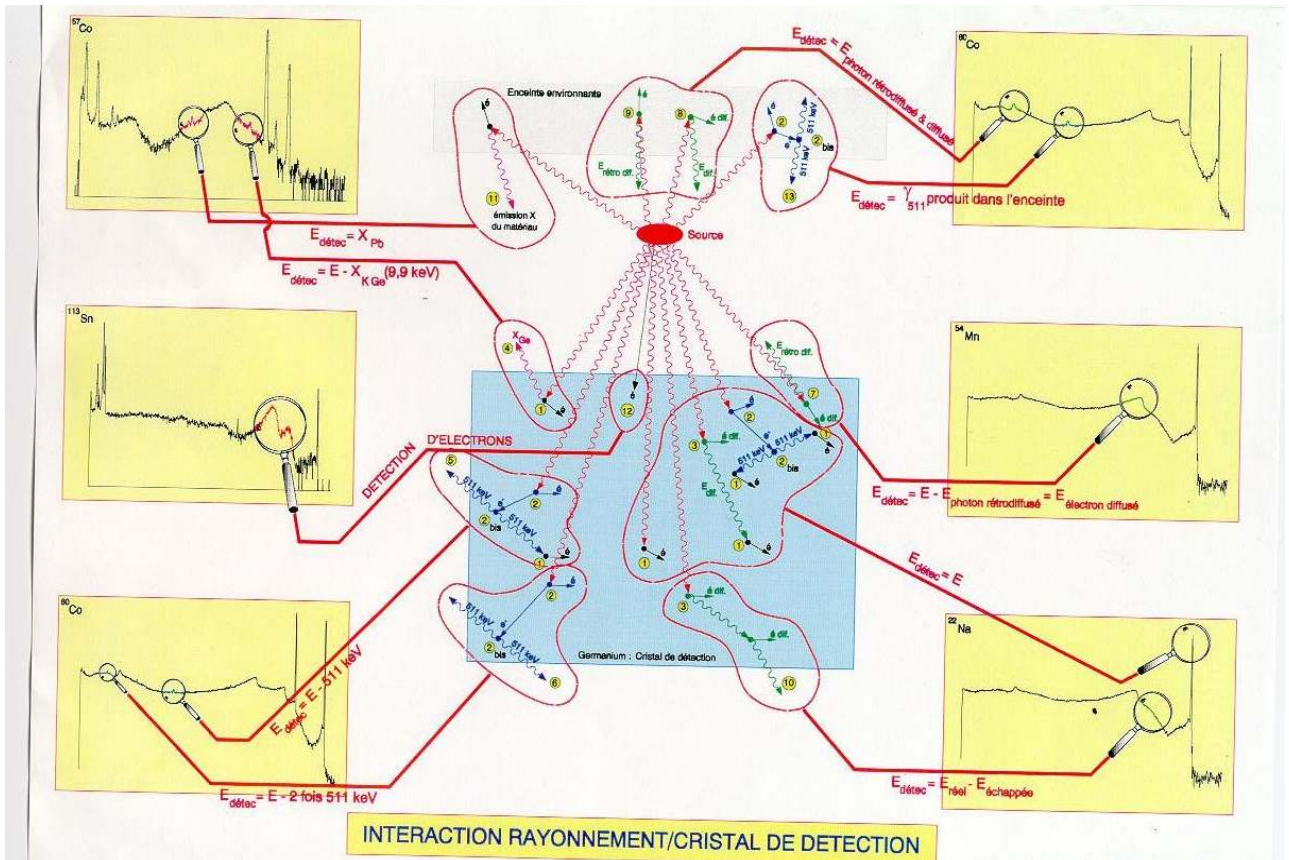


Figure 23 Photon-Detector crystal interactions

1	Photoelectric Absorption] →	$E' = E$
2	Pair Production		
2 bis	Annihilation		
3	Compton Effect		
4	Escape phenomena	→	$E' = E - X_{Ge}$
5	Single Escape phenomena] →	$E' = E - m_0C^2$ $E' = E - m_0C^2$
6	Double Escape phenomena		
7	Compton Effect: Backscattering with escape of the scattered photon	→	$E' = E - E_{diff}$
8	Exterior Compton Effect : Detection of the scattered photon	→	Energy distribution
9	Exterior Compton Effect : Backscattering		
10	Compton Effect with partial collection of the incident photon	→	$E' = E - E_{escaped}$

- 11 X Fluorescence of the surrounding shielding
- 11 Detection of the electrons emitted by the source
- 13 Detection of the 511 keV photons: Pair production in the shielding (if $E > 1,022 \text{ MeV}$)

Figure 23 above shows the different possible interactions that may take place between the incoming photons and the detector material.

5.6. Spectrum components

5.6.1. Full energy peak (FEP)

A full energy peak is the result of detecting full energy events.

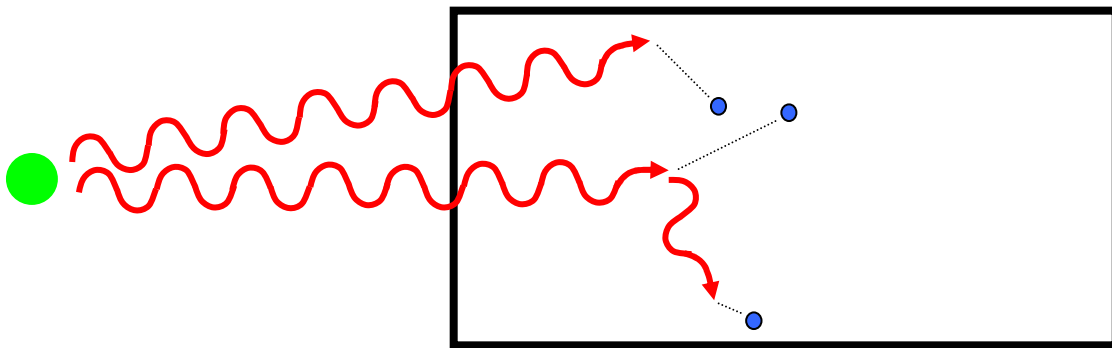


Figure 24 full energy events

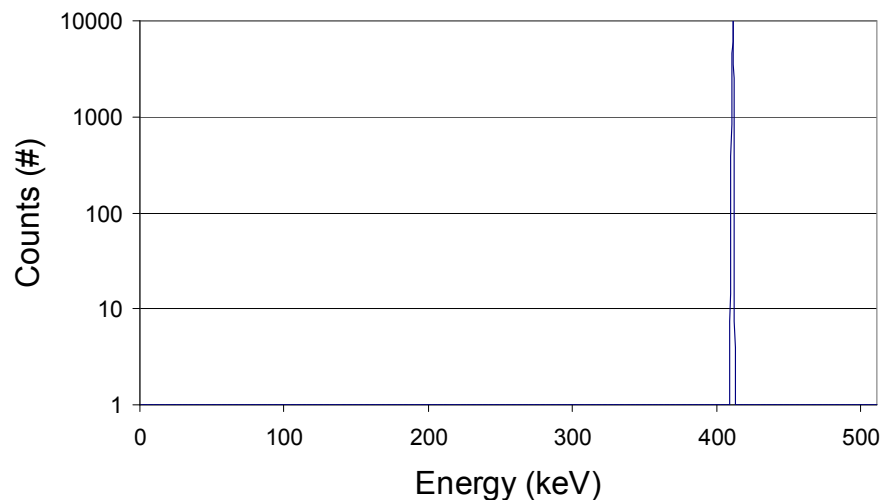


Figure 25 full energy peak

5.6.2. Compton Continuum and Compton edge

The scattered gamma-ray will inevitably escape from the detector, taking with it the remaining gamma-ray energy. The detector response to Compton interactions will, therefore, mirror the curve shown in figure 27 and the corresponding gamma-ray spectrum would exhibit the characteristic Compton continuum extending from zero energy up to the Compton edge illustrated on the right hand side of Figure 27. There would be no Compton scattering contribution to the full energy peak.

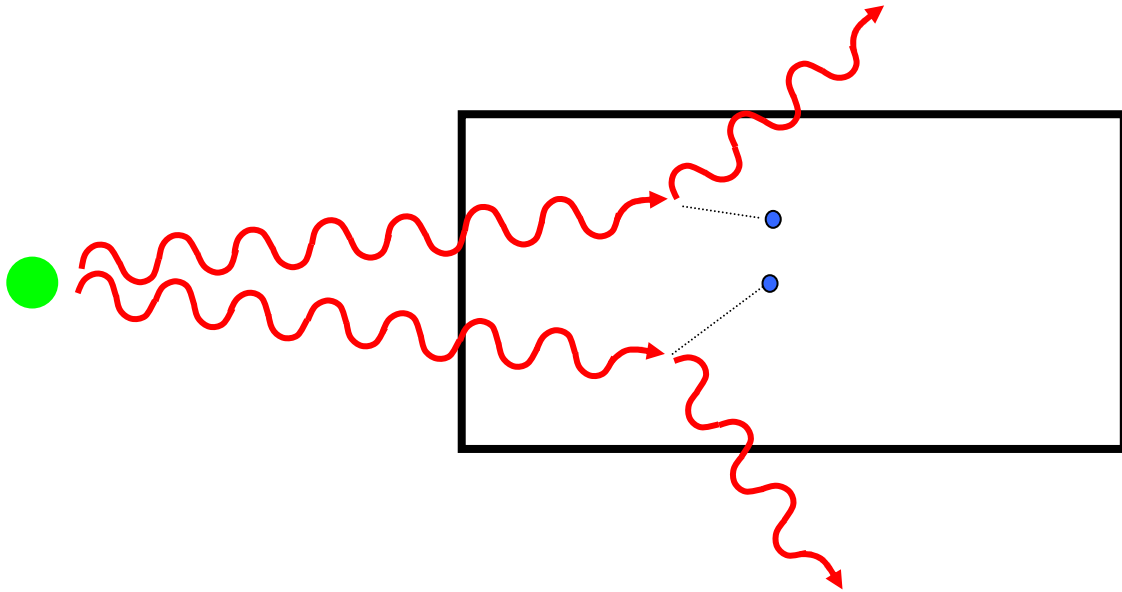


Figure 26 The scattered gamma-rays escape from the detector

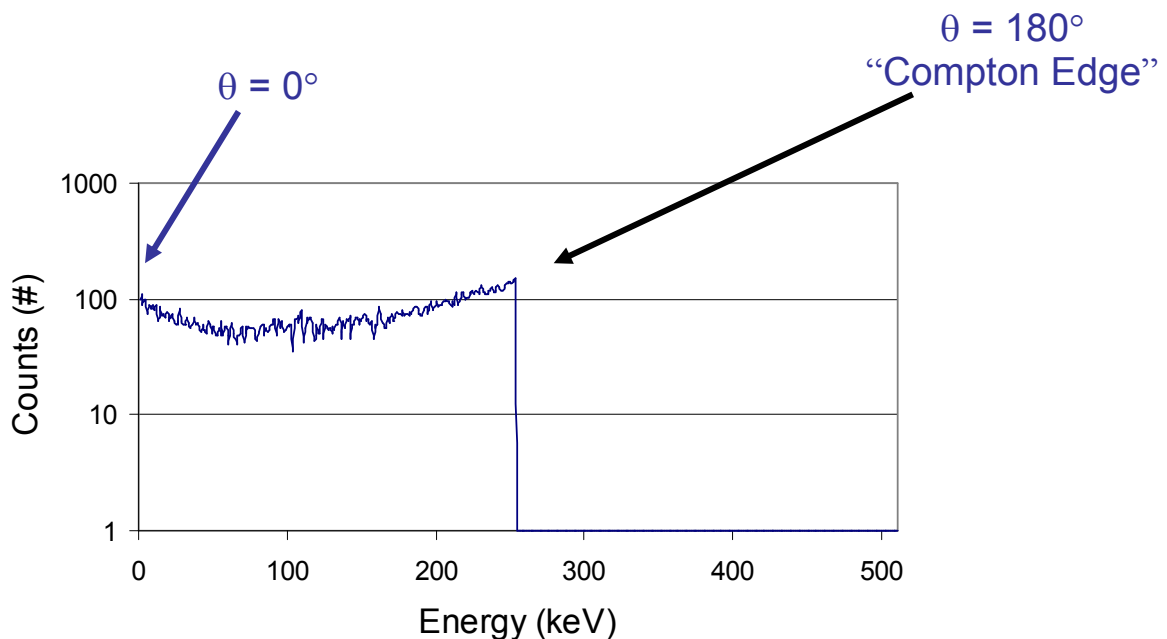


Figure 27 Compton Edge and continuum

Example of Gold Isotope

^{198}Au (2.6 day) Decay Scheme

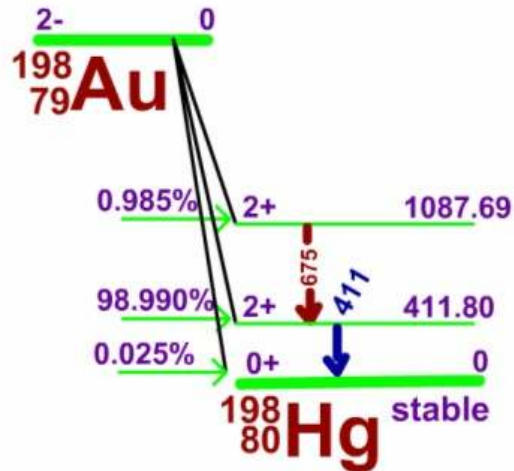


Figure 28 HPGe Gamma Spectrum of ^{198}Au

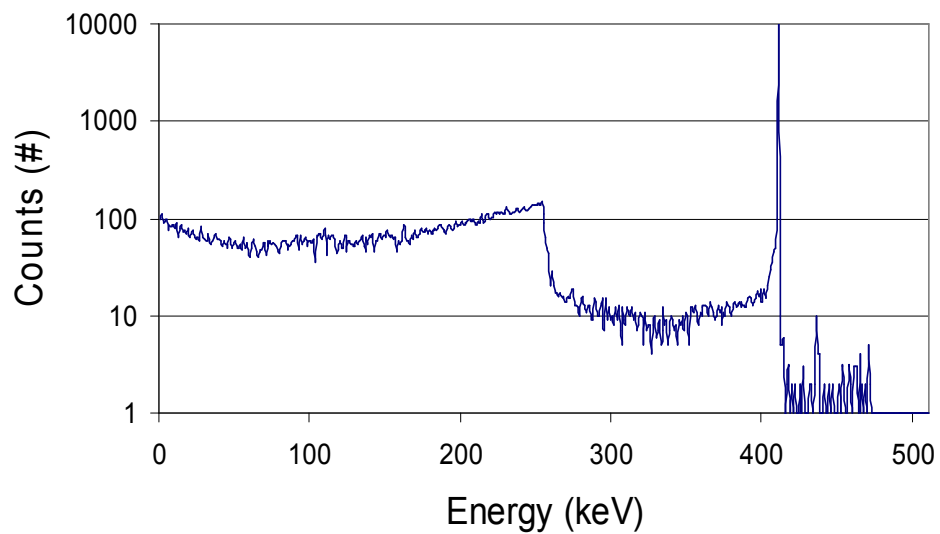


Figure 29

5.7. Other components of spectrum

- Sum peak
- Single escape peak
- Double escape peak
- Annihilation Peak

5.7.1. Sum Peak

Let us look at ^{60}Co . It is an activation product commonly used for energy calibrations as well as QA/QC.

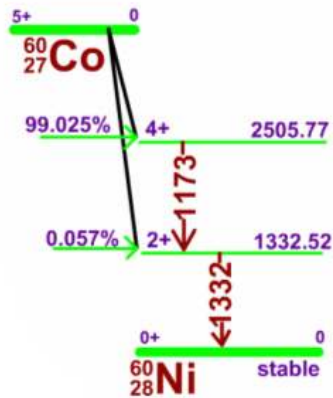


Figure 30 ^{60}Co Decay Scheme

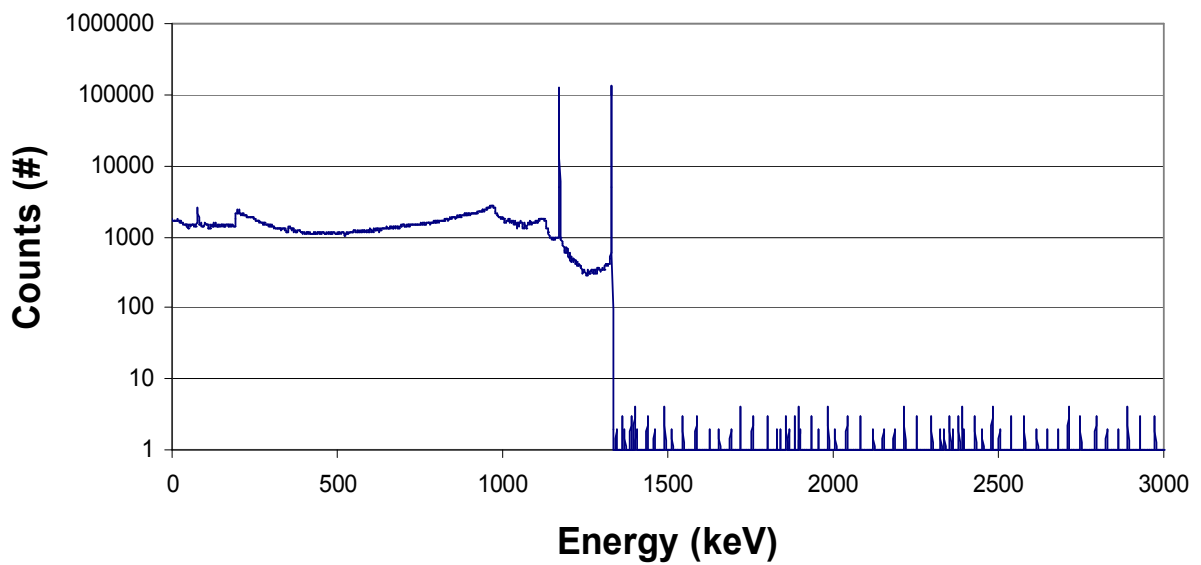


Figure 31 Expected Spectra Based on Decay

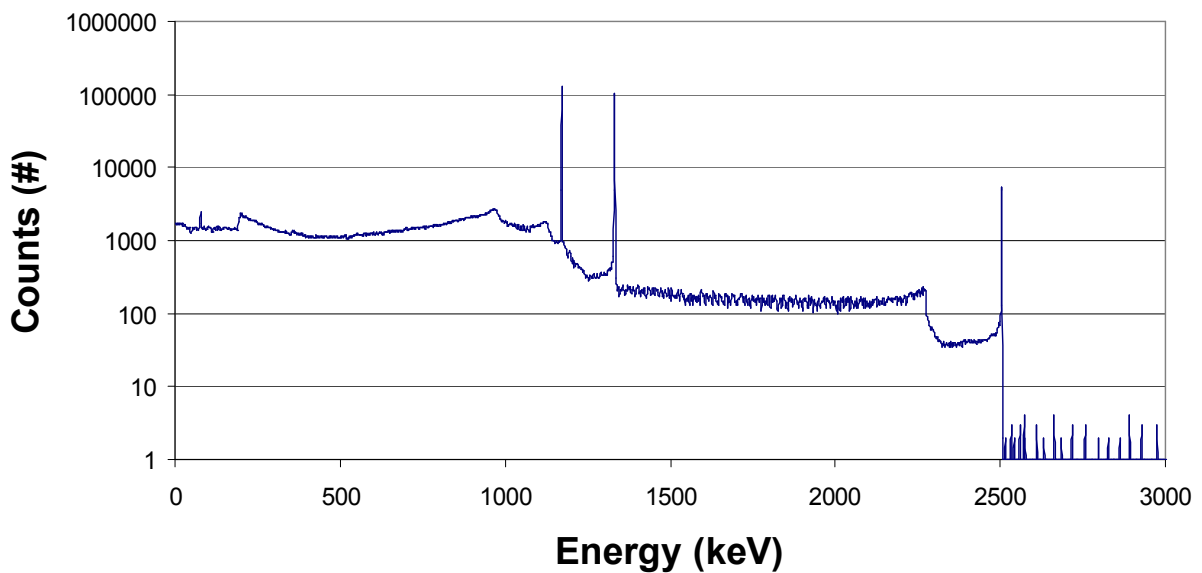


Figure 32 Actual Spectra

Reason

When an isotope emits more than one photon in a single decay process, there is a chance that both will deposit energy in the detector. When this happens, a **sum peak** can be observed. Sum peaks can also be observed in samples containing isotopes that emit only one photon per decay. This is generally only true for high activity samples.

5.7.2. Single Escape Peaks

Because the detector volume is finite, there is a possibility that one of the annihilation photons created from pair production events in the detector can escape. When this happens, a peak can be observed at energy equal to the FEP - 511 keV.

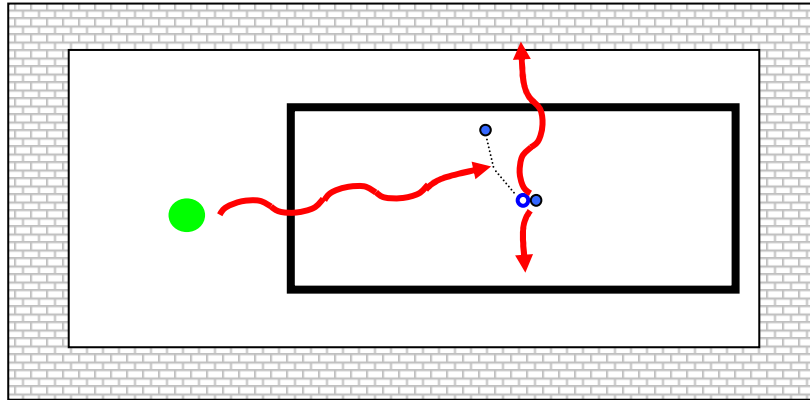


Figure 33

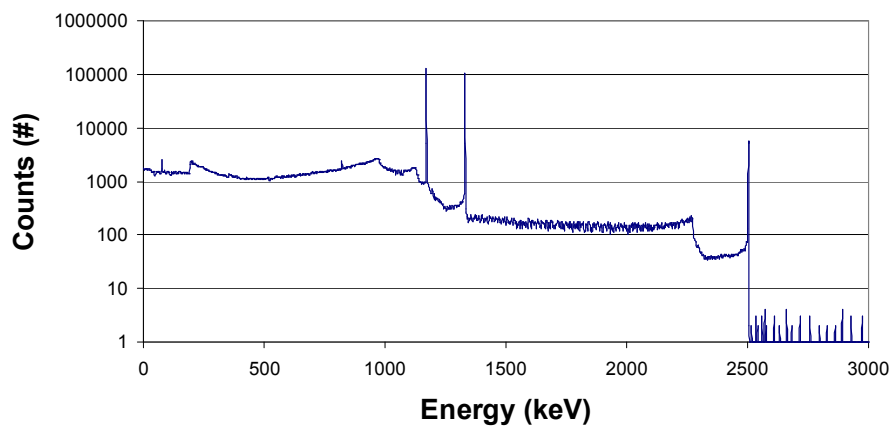


Figure 34 Single Escape Peak

5.7.3. Double Escape Peak

If both annihilation photons exit the detector, then a peak can be observed at FEP – 1022 keV.

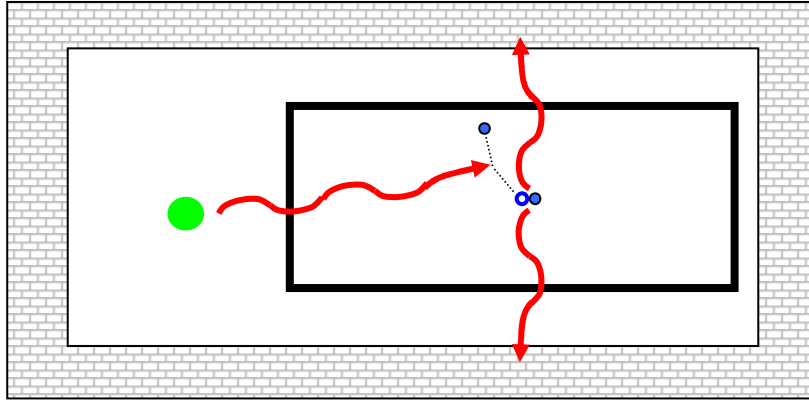


Figure 35

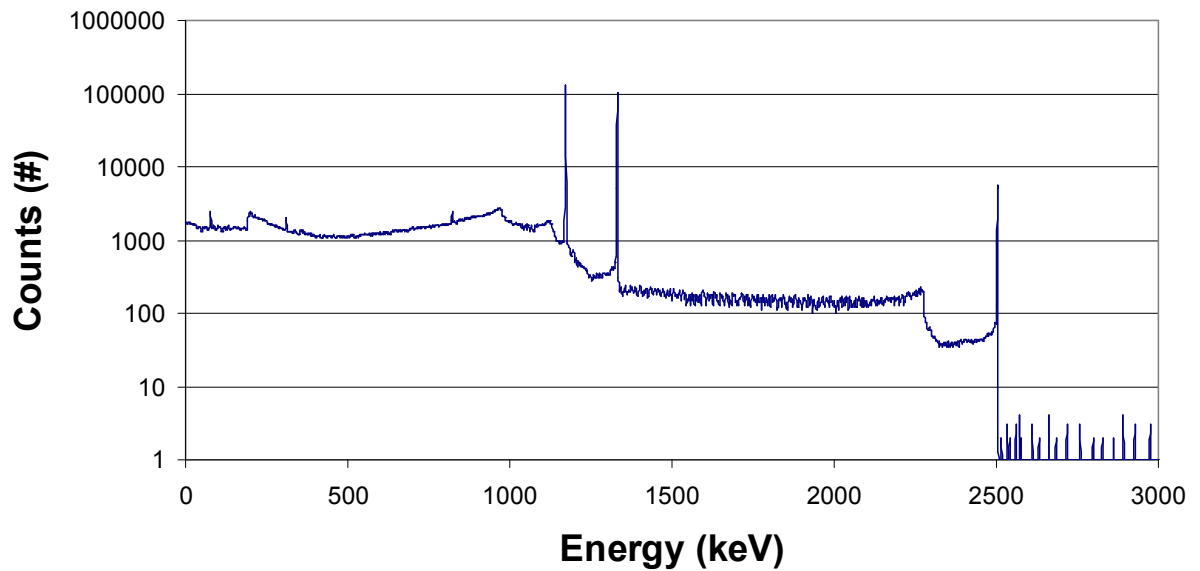


Figure 36 Double Escape Peak

5.7.4. Annihilation Peak

An interesting situation can develop when a high energy photon ($E > 1022 \text{ keV}$) starts the sequence: Some of the annihilation photons can escape from the detector. No distinction can be made between the two photons produced on annihilation of the positron, and the following cases can be distinguished:

- both annihilation photons are totally absorbed: total energy event;
- only one annihilation photon is totally absorbed, while the other escapes from the detector: **single escape** event; and
- none of the two photons are totally absorbed: **double escape** event.

If a pair production event occurs in the surrounding shielding material, there is possibility that one of the two annihilation photons will deposit energy into the detector. When this happens, an *annihilation peak* can be observed at 511 keV.

•

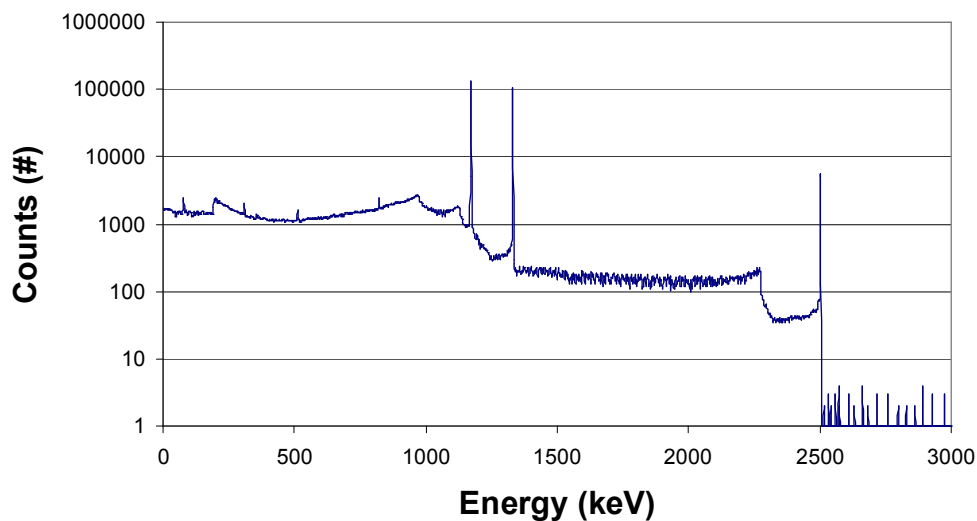


Figure 37

The various spectrum components are illustrated in figure 38

Components of Spectrum

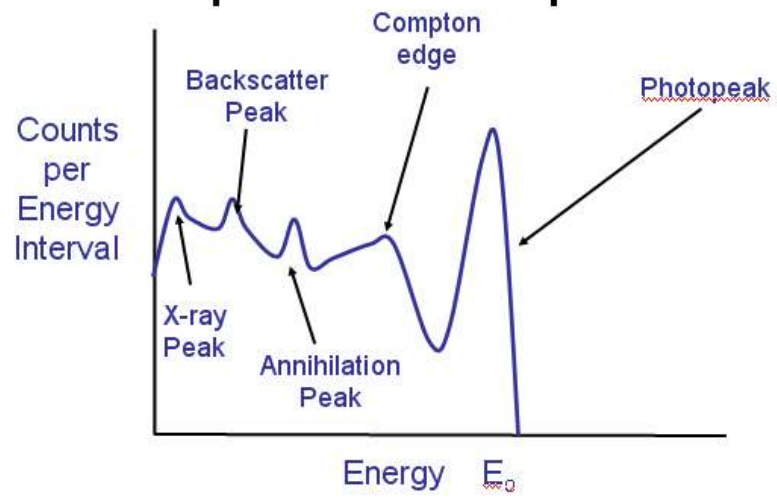


Figure 38

5.8. Influence of the surrounding material on the detector

Emitted gamma photons may also interact with the surrounding material.

5.8.1. Backscatter peak

Here we are looking at the scattered photon off the surrounding lead. The peak appears at the energy (few hundred keV) from a photon scattered back at $\sim 180^\circ$.

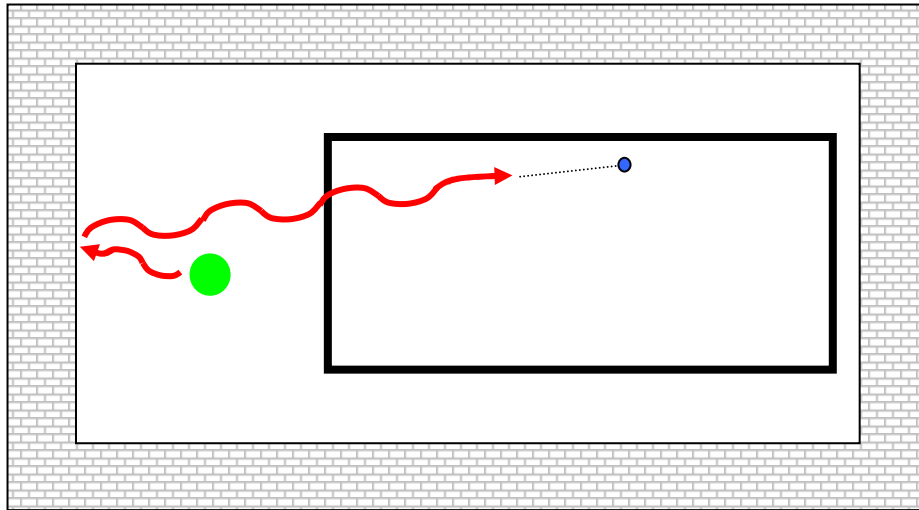


Figure 39

5.8.2. Characteristic X-rays

Photons emitted by the source ionize the surrounding material. Characteristic x-rays are emitted as electron shell transitions take place to fill the lower empty shell.

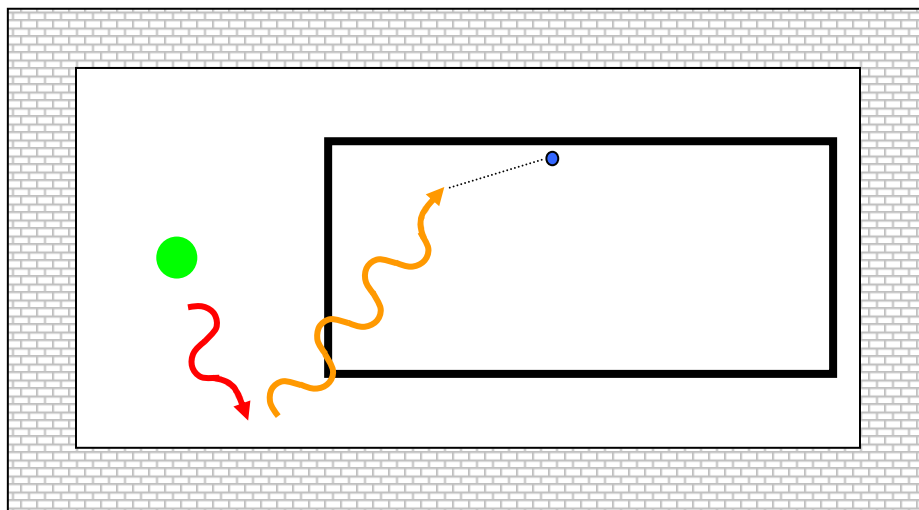


Figure 40

To eliminate the peaks resulting from the characteristic x-rays, *graded shields* are introduced. Typically, cadmium sheets are placed on the lead followed by copper on the cadmium. Other materials can be used *as long as you go from high-to-low Z materials*. Copper and cadmium are used because both are malleable and cheap.

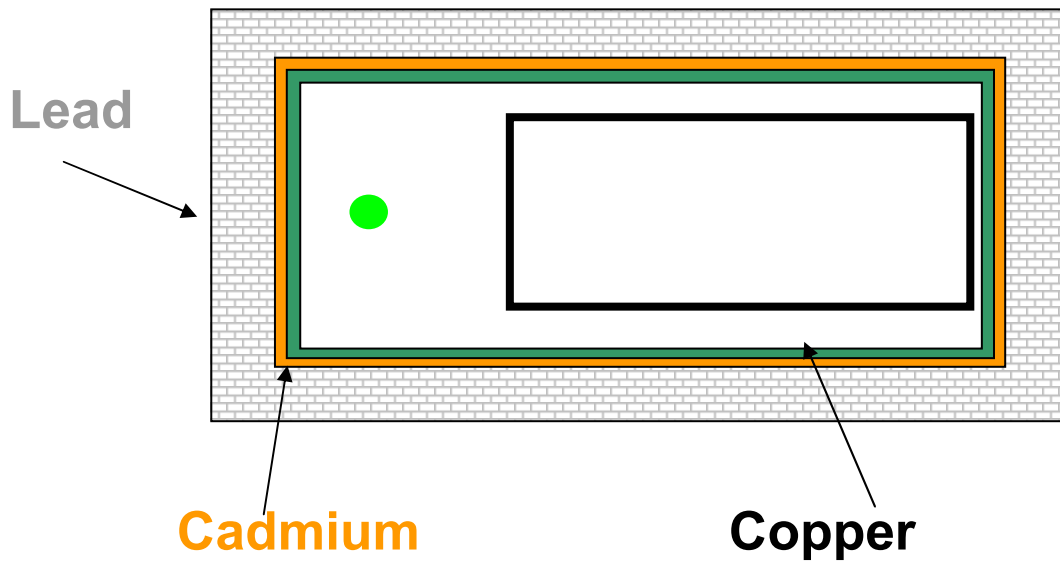


Figure 41

Table 2: Fluorescence X line from lead

Energy (KeV)	Intensity (%)	Line
74,815	$10,6 \pm 0,2$	$K_{\alpha 2}$
77,108	$17,8 \pm 0,3$	$K_{\alpha 1}$
86,834	$2,07 \pm 0,03$	$K_{\beta 3}$
87,343	$3,94 \pm 0,07$	$K_{\beta 1}$
89,8	$1,67 \pm 0,03$	$K_{\beta 2}$

Influence of Surrounding Materials on Detector Response

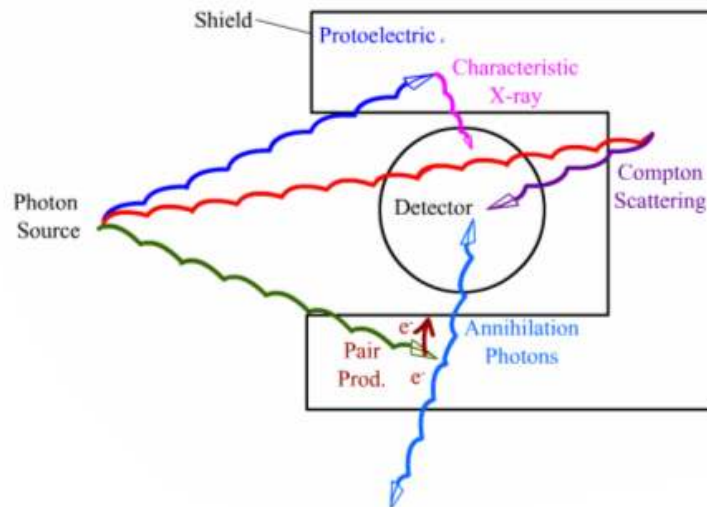


Figure 42

Influence of Surrounding Materials on Detector Response

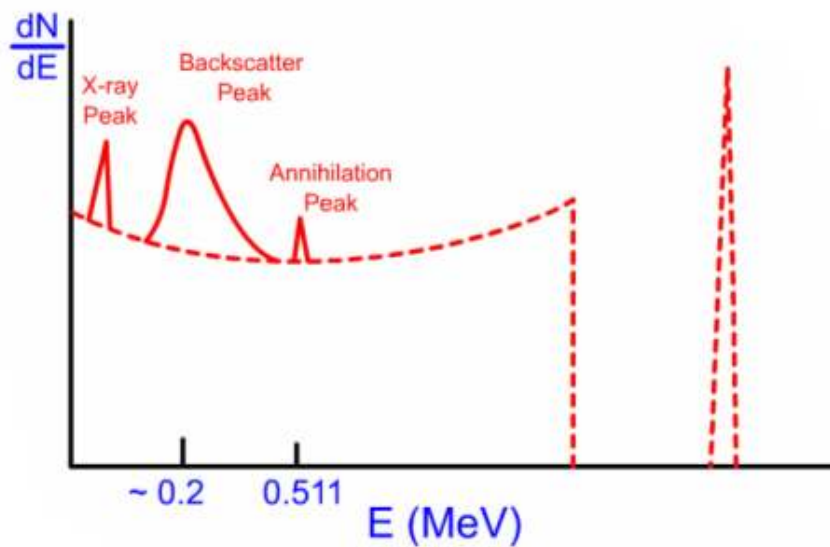


Figure 43

6. SPECTROMETER CALIBRATION AND SPECTRUM ANALYSIS

6.1. Spectral analysis software

The Basic Spectroscopy Software usually includes a set of basic analysis algorithms for processing of gamma spectra acquired from any type of gamma detector, see table 3.

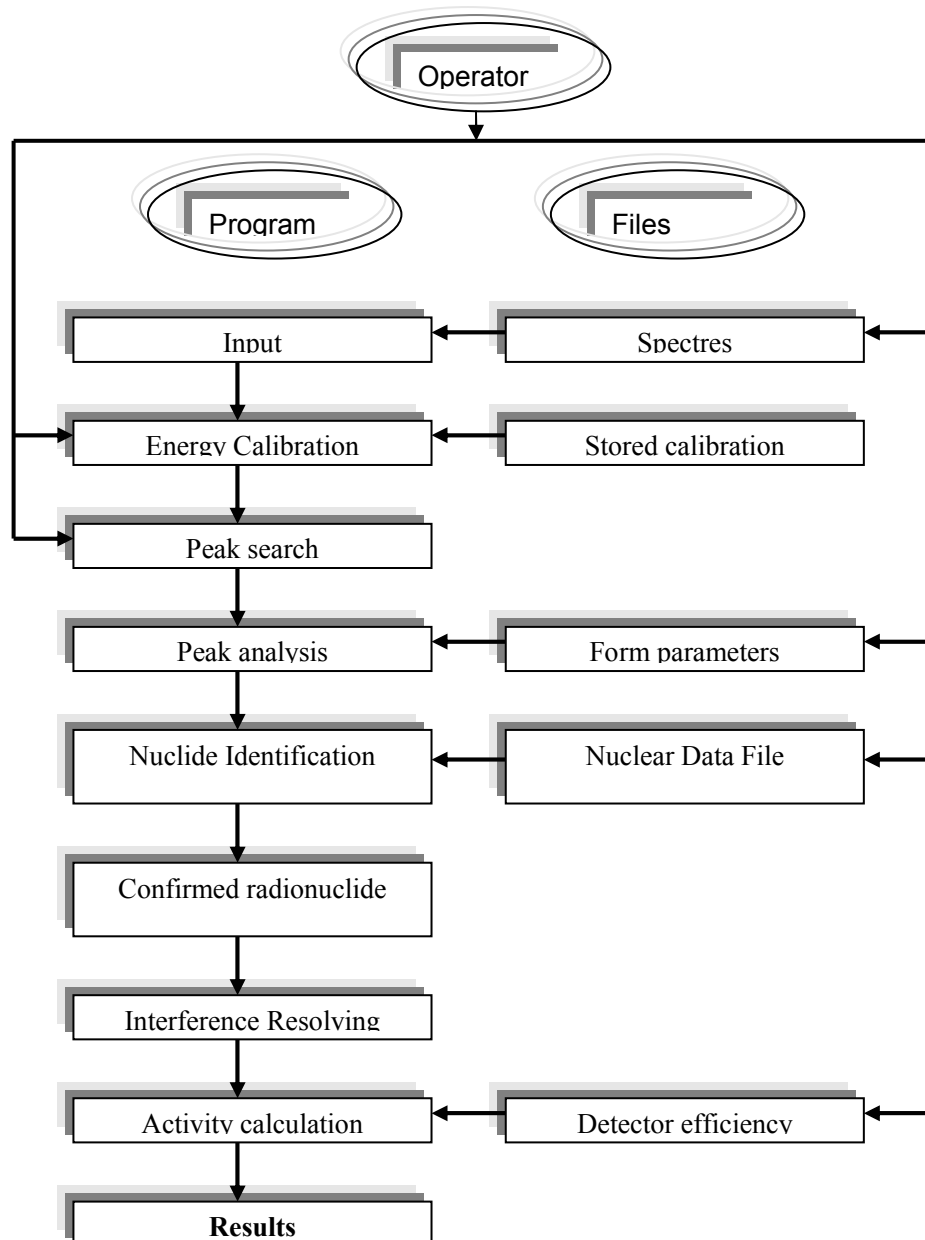


Table 3 General steps for spectrum analysis γ

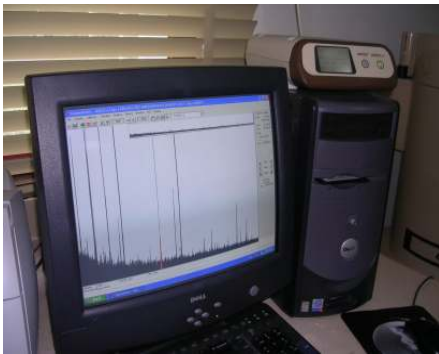
Figure 44

6.2. System calibration and characterization

The system calibration consists of the following steps

- | | |
|----------------------------------|--|
| ☞ Energy Calibration | $E = \text{function of Channel \#}$ |
| ☞ Efficiency Curve determination | $\text{Ln}(\text{eff}) = \text{function of Ln}(E)$ |
| ☞ Resolution Calibration | $(\text{FWHM})^2 = \text{function of Energy}$ |

6.2.1. Energy calibration



A least squares fit to two or more pairs of energy and channel number points calibrates the ADC range in energy level (eV, keV or MeV). Second-order calibration is also provided to allow more precise calibration over the entire range. Calibration in units of time (microseconds, milliseconds, or seconds) is included for multichannel scaling.

A lot of effort (and money) is applied to retain the linear relationship between the amount of energy transferred to the detector (E , keV), and the height of the electronic pulse resulting from it; and also to the number of the channel in which this pulse is stored (K). In general:

$$E = m \cdot K + c$$

where: m is the linear energy scale (keV/channel) of the MCA; and c is the intercept (which is usually set as close as possible to zero).

An energy scale of 0,3 keV/channel is frequently used in high-energy gamma spectrometry to cover the energy region up to 2400 keV for a 8K MCA system (i.e. 8192 channels). A scale of 0,25 keV/channel would probably utilize the resolution of the detector better, but will only cover the region up to about 2000 keV. For low-energy spectrometry an energy scale of 0,05 keV/channel would often be adequate. It is not possible to calculate accurate values for m and c . The analyst is therefore forced to determine these values empirically.

An active sample containing a number of nuclides that emit photons covering the desired energy range, is counted with the detector to be calibrated. The true positions (channel number) of every full-energy peak is determined accurately, and the energy of the photon plotted against channel number. A single nuclide emitting a sufficient number of photons (e.g. ^{152}Eu) can also be used.

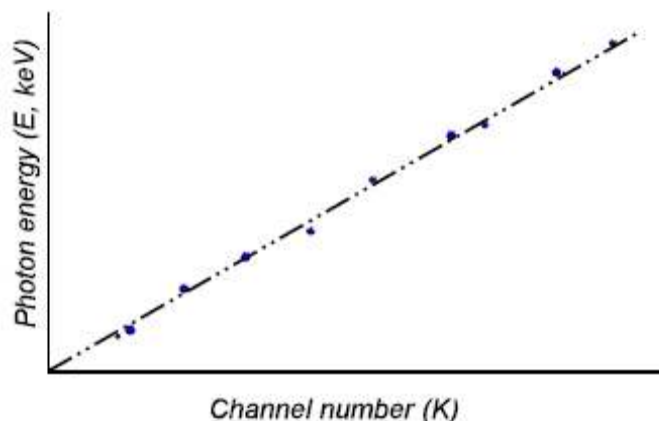


Figure 45

This curve can be used to determine the energy of a photon that is responsible for an unknown peak in the spectrum. The software usually determines the values of m and c by fitting a linear function to all the available points. The parameters describing the FWHM and tailing of the peaks as function of photon energy are also determined as a part of this action. These values are stored, and recalled whenever an unknown pulse height spectrum is to be analyzed. The following must be considered:

- (a) The electronic modules are not absolutely stable under all conditions, and one finds that the values of m and c drift slightly with time. This requires frequent energy calibration.
- (b) The MCA can, however, accept only one set of values as valid at any particular time: the results from the previous energy calibration.
- (c) The analyst must therefore establish empirically the rate of drift for every detector system, and decide on a energy calibration frequency that would generate new calibration parameters before the expected error becomes too large.

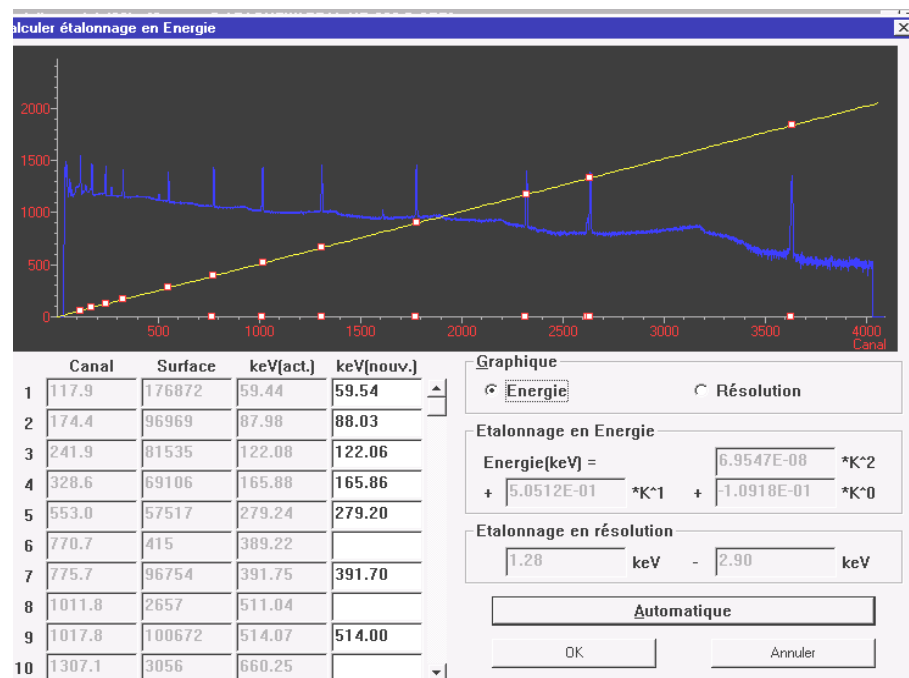


Figure 46 Energy Calibration Example using Genie-2000 software (Canberra)

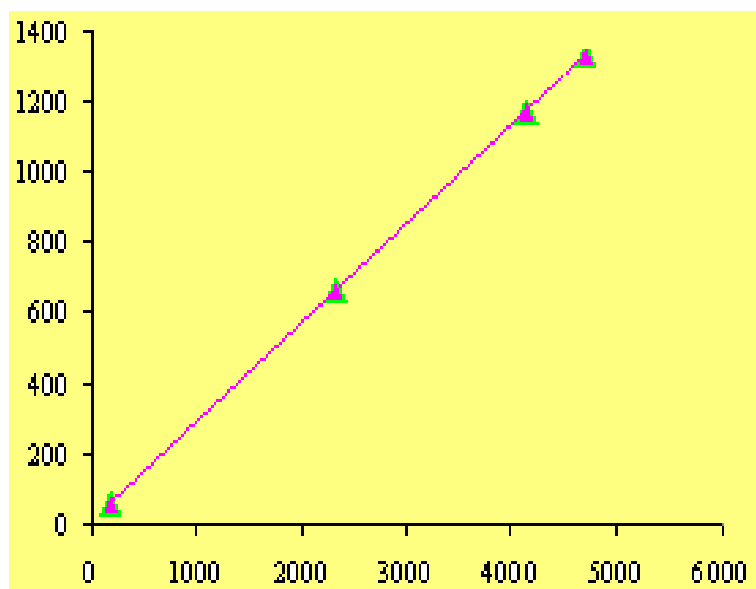


Figure 47 Energy Calibration curve for 30 % efficiency HPGe

6.2.2. Peak form and energy resolution

If a single photon nuclide is counted (e.g. ^{137}Cs at 661 keV), all the pulses from the full energy events should theoretically have the same height. In practice it is found that the height values are spread over a narrow band of channels of the MCA. The **full width at half the maximum height (FWHM)** of a peak in the pulse height spectrum is used as a measure of this energy "spread". This is a quantitative indication of the expected **resolution** of a detector; i.e. its ability to distinguish between two peaks of equal size that are close together. It can be expressed as a relative value (%) or an absolute (keV) value. Typical values (e.g. at 661 keV) are about 50 keV for NaI scintillation and 1,5 keV for HPGe detectors. The peak form can be represented as largely "normal" (i.e. Gaussian distribution) with some "tailing" at the lower energy region. This distortion is normally expressed in terms of a **tailing parameter**.

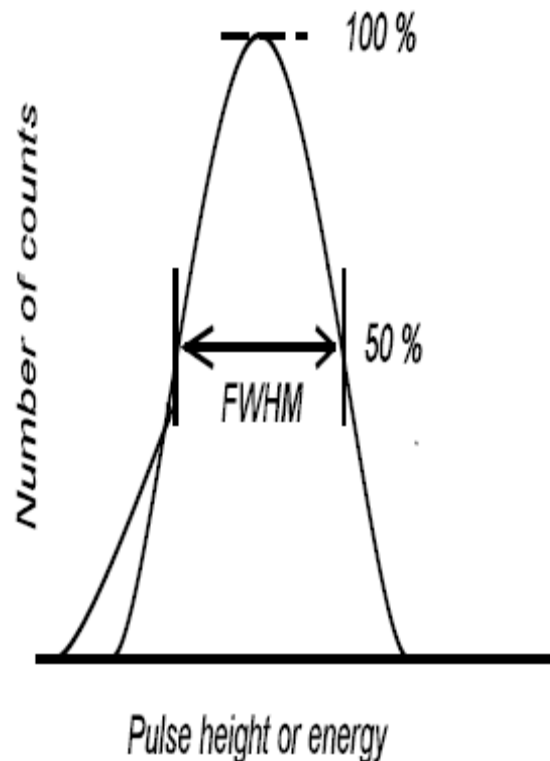


Figure 48

Both the FWHM value and tailing parameter increase with the energy of the photon, and both are dependent on intrinsic properties of the detector, the type and setting of the electronic modules being used, and the layout of the cables. It requires rather complex data processing to establish these relationships, but

these facilities are normally built into the software. The analyst needs only to count one or more calibration samples that cover the energy region to be investigated. These data are then used by the software to establish the mathematical relations that typically is in the form:

$$FWHM = a + b \cdot \sqrt{E_\gamma} \quad \text{and} \quad Tail = c + d \cdot E_\gamma$$

These relations are then used by the system to calculate the expected FWHM and tailing parameter at any photon energy of interest.

For automatic peak analysis, it is necessary to tell the computer what the peak width as a function of energy is. In general, this is known as the Full Width at Half Maximum (FWHM). The procedure is much the same as for the energy calibration. Indeed, it may be performed simultaneously. Resolution calibration is generally given in the following form:

$$R(x) = \sqrt{\alpha + \beta \cdot x}$$

Where:

X is the peak maximum channel number,
 α and β unknown constants

Figure 51 shows a FWHM calibration curve for a HPGe. A best-fit straight line has been drawn through the points.

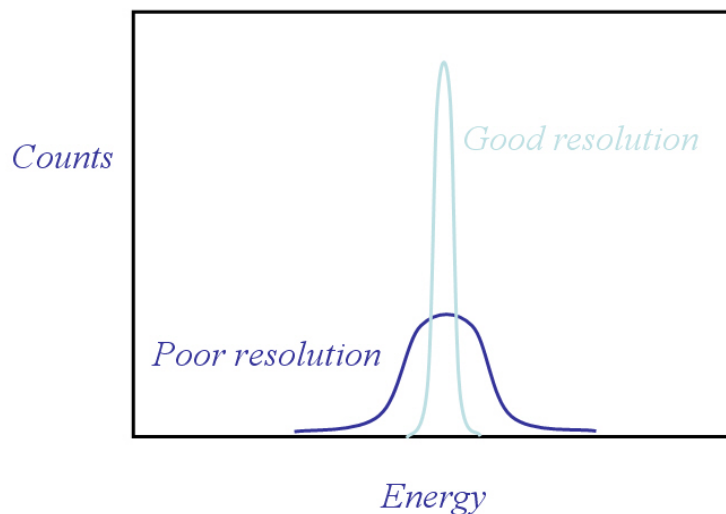


Figure 49 Energy Resolution

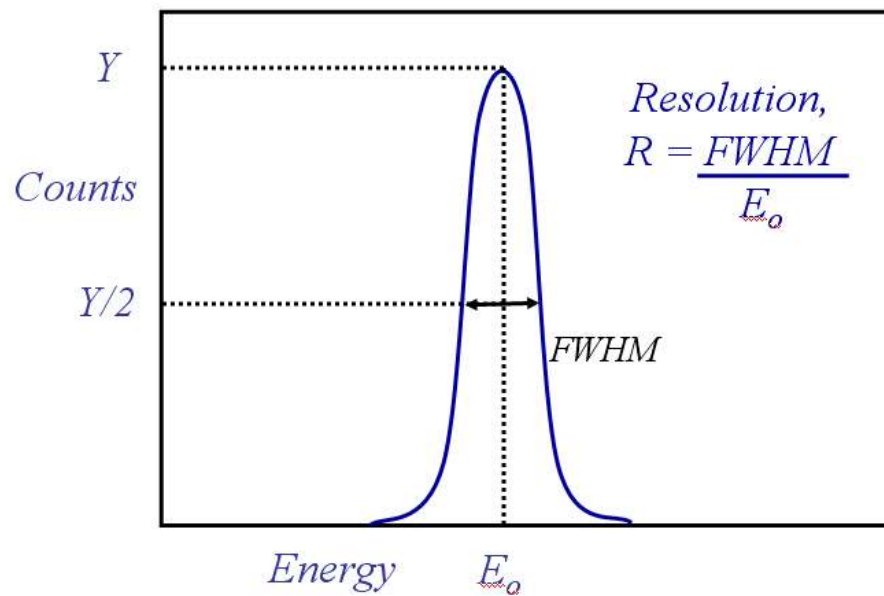


Figure 50 Energy Resolution

$$(FWHM)^2 = 0.0022 (E) + 0.4063$$

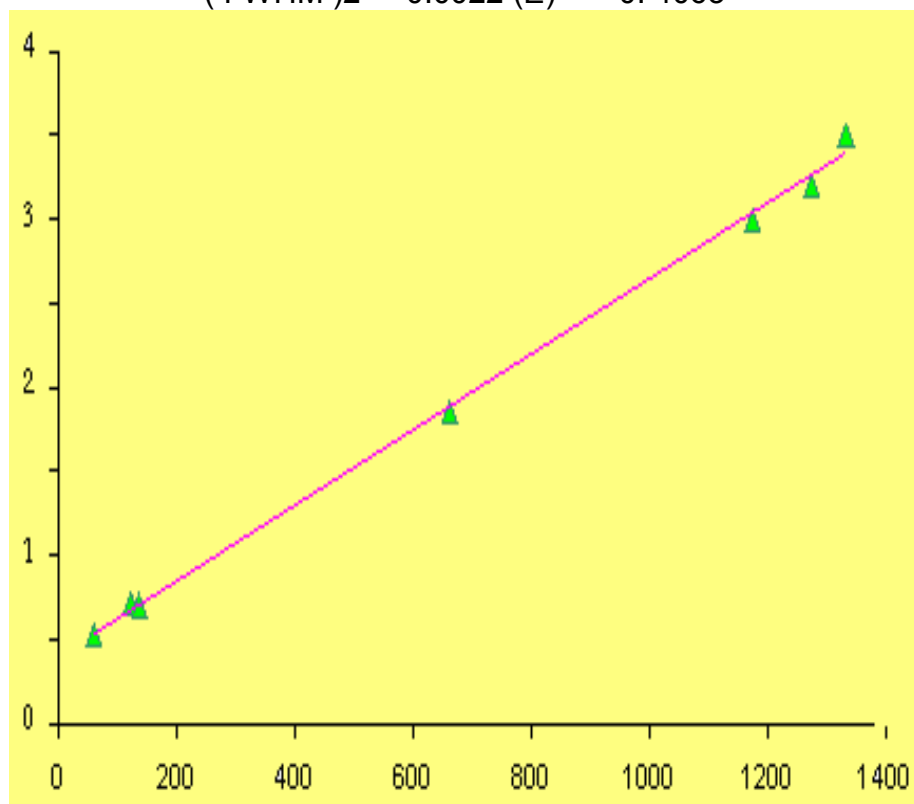


Figure 51 calibration curve for a 30 % HPGe

Germanium Detectors – Loss of Resolution

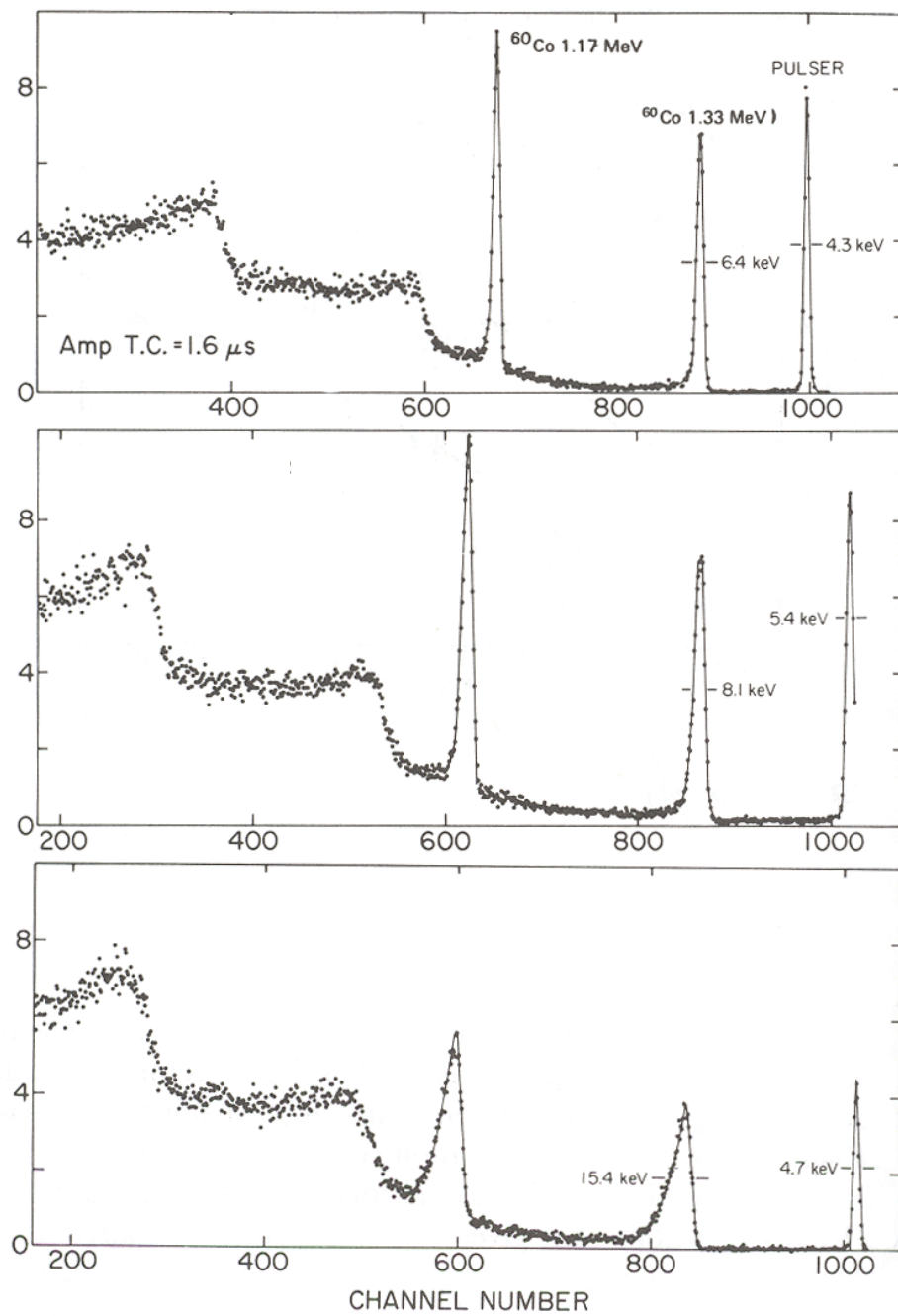


Figure 52 The deterioration of a Ge gamma-spectrum

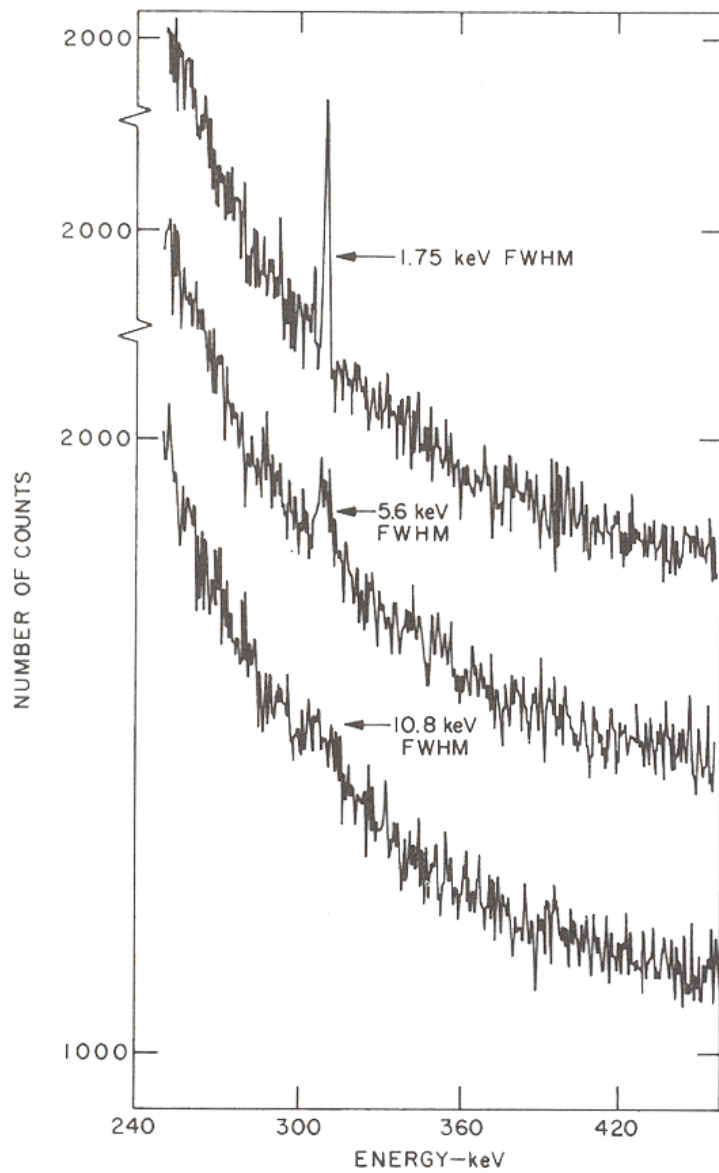


Figure 53 Germanium detectors – loss of resolution

6.2.3 Efficiency calibration

The full-energy peak efficiency is the parameter the most significant in gamma-ray spectrometry, it is denoted by ε . It is defined as the fraction of the photons of a particular energy emitted by a source, that contributes to the corresponding full-energy peak observed in the pulse height spectrum, so that:

$$P = \varepsilon \cdot Y \cdot A$$

where: P is the counting rate in the full-energy peak observed in the pulse height spectrum, ε is the counting efficiency; Y is the photon yield (i.e. fraction of decay events that result in the emission of a photon of the relevant energy); and A is the activity of the source in Bq.

The value for efficiency is dependent on the geometry of the sample – size, density, and distance from detector. For the detectors used in gamma analysis, efficiency varies significantly as a function of these parameters (see Figure 54). Therefore, each counting geometry requires an efficiency calibration, using a known standard in the same geometry which includes multiple energies.

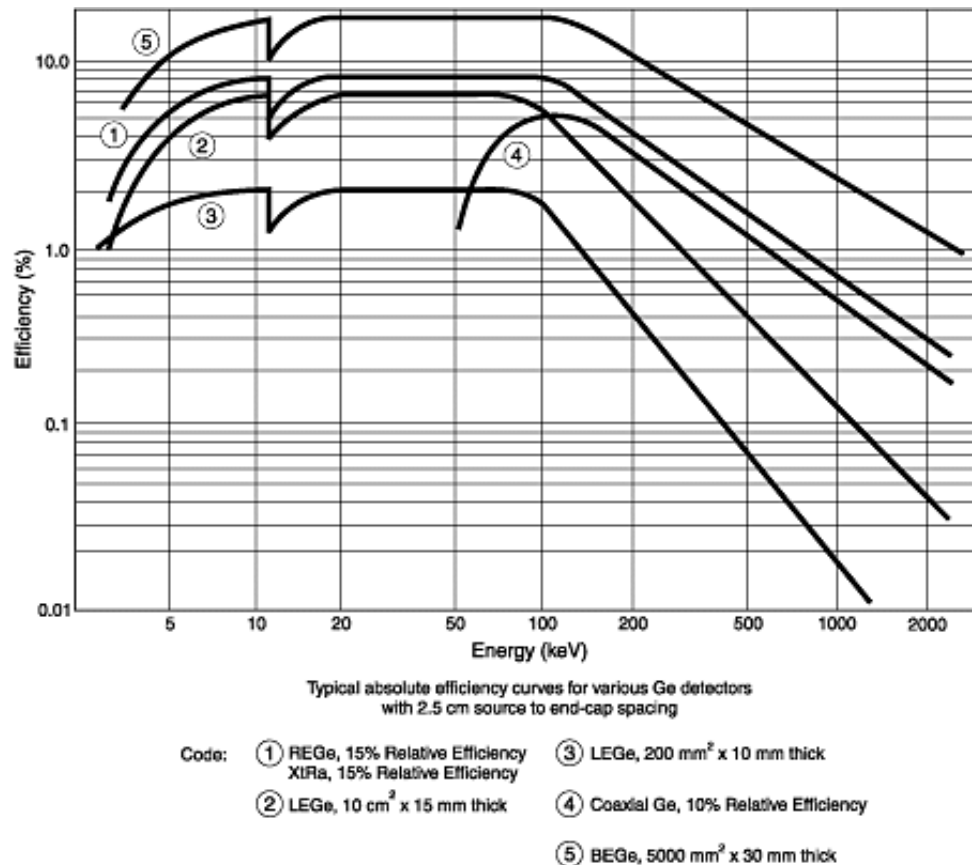


Figure 54

A series of data pairs of efficiency vs. energy are generated from the relationship:

$$\text{Efficiency} = \frac{\text{Net Area}}{(\text{Live Time})(\text{Activity})(\text{Yield})}$$

Where Activity is the strength (in Bq) of the standard source (at collection time) at the given energy, yield is the branching ratio fraction and live time is the actual ADC live time in seconds.

Usually one needs to correct for source decay using the following the formula:

$$A(\text{at count time}) = A(\text{at certificate time})e^{-\frac{\ln(2) \times \text{Decay Time}}{\text{Half Life}}}$$

Where decay time and half-life are in the same units (seconds, minutes, hours, or years).

From the series of data pairs, a curve of efficiency versus energy is generated, with the user having a choice of fitting paradigms. Thus, the software can calculate efficiency at any energy in the calibrated energy range when analyzing an unknown spectrum.

The value of the counting efficiency is determined by the following factors:

- Attenuation of photons inside the source due to absorption in the sample material: This effect can be ignored if the density of the material is less than 1 g.cm⁻³ and the thickness of the sample layer is less than 2 cm.
- Attenuation of photons in the canning material over the entrance face of the detector: The magnitude of this effect depends on the thickness and composition of the entrance window, and on the energy of the photons. It can be ignored for aluminum windows if the energy exceeds 100 keV.
- The fraction of the photons emitted by the source that hits the effective volume of the detector: This is determined by the form and dimensions of the sample and the detector, and the effective distance between the two.
- The fraction of the photons hitting the detector that contributes to the full-energy peak: This is determined by the physical size of the detector and on the photon energy.

It is in practice not possible to calculate the efficiency values at different energy values for all the geometric arrangements that are used in the laboratory. It is

therefore standard practice to prepare calibration samples and determine the efficiency of the detector empirically. These calibration samples must meet the following requirements:

- They must be similar to the field samples (that are to be analyzed) in every respect: matrix composition, physical form and dimensions. It must be possible later to put the field samples in exactly the same position relative to the detector.
- They must contain a number of nuclides (or a nuclide emitting a large number of different energy photons) to cover the energy region that is of interest.
- Their activity values must be known accurately, and traceable to a national standard.
- They must be mechanically robust (to prevent contamination of a detector) and chemically stable (to eliminate segregation of the active component from the matrix)

It is normally not possible to purchase calibration standards that satisfy all these requirements, and the analyst is compelled to prepare it in the laboratory. It demands a lot of innovation; and often the analyst must compromise because of practical problems. The problem can be simplified somewhat if a limited number of geometric arrangements are defined, so that the number of calibration samples can be reduced. Special rigs can also be designed to ensure that samples and standards are always placed in the same position.

Another issue to consider is the best model for expressing the mathematical relationship between detector efficiency and photon energy values. The following models are often used:

$$\ln \epsilon = a_0 + a_1 \cdot (\ln E_\gamma)^1 + a_2 \cdot (\ln E_\gamma)^2 + a_3 \cdot (\ln E_\gamma)^3 + \dots$$

and

$$\ln \epsilon = a_0 + a_1 \cdot \left(\frac{1}{E_\gamma} \right)^1 + a_2 \cdot \left(\frac{1}{E_\gamma} \right)^2 + a_3 \cdot \left(\frac{1}{E_\gamma} \right)^3 + \dots$$

The software normally offers a number of models, and allows the analyst to decide how many terms to include in the polynomial expression. These options should be selected with care, bearing in mind a number of problems that might arise:

- Only a limited number of nuclides are available as certified standards, and it is difficult to cover the full energy range.
 - The values change quite rapidly around the "knee" of the curve. Serious errors can be introduced if this curvature and the region below it are not adequately covered.

- On the high energy side, only ^{152}Eu at 1407 keV and ^{88}Y at 1836 keV are available. If the analyst is using an energy scale of 0,6 keV/channel with ^{152}Eu for calibration, the experimental information covers only the region up to channel 2400. The region between channels 2400 and 4000 is not covered, and efficiency values for peaks found there must be calculated by extrapolation. Serious errors can be introduced, especially by the high-order terms in the polynomial which have a dramatic effect in the extrapolation.
- (b) If the nuclide decays *via* a gamma cascade (e.g. ^{60}Co), there is a possibility of having sum coincidences of the two photons. The counting rates in the two single full energy peaks and the sum peak are given by:

$$P_1 = \epsilon_1 \cdot A$$

$$P_2 = \epsilon_2 \cdot A$$

$$P_{12} = \epsilon_1 \cdot \epsilon_2 \cdot A$$

All the pulses appearing in the sum-peak, have been "removed" from the two single peaks. The measured peak areas and efficiency values are too low. This effect can be reduced by counting the sample at a greater distance (i.e. by reducing the values of ϵ_1 and ϵ_2); but this has a negative effect on the sensitivity of the method.

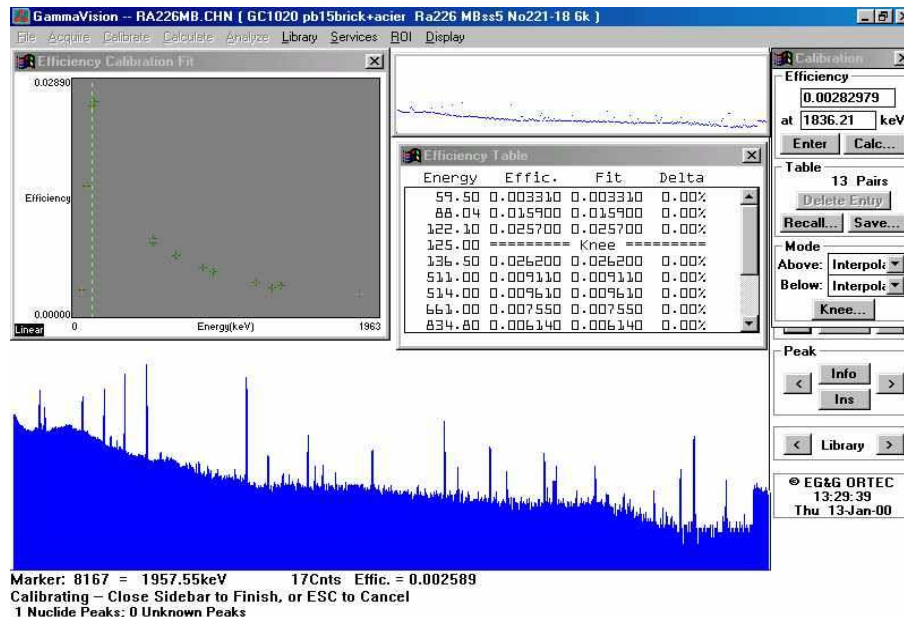


Figure 55 Energy Calibration Example using GammaVision software (Ortec)

LeGe detector : Efficiency Calibration for bulk geometry
(Cylindrical box 0.282L 4M HCl)

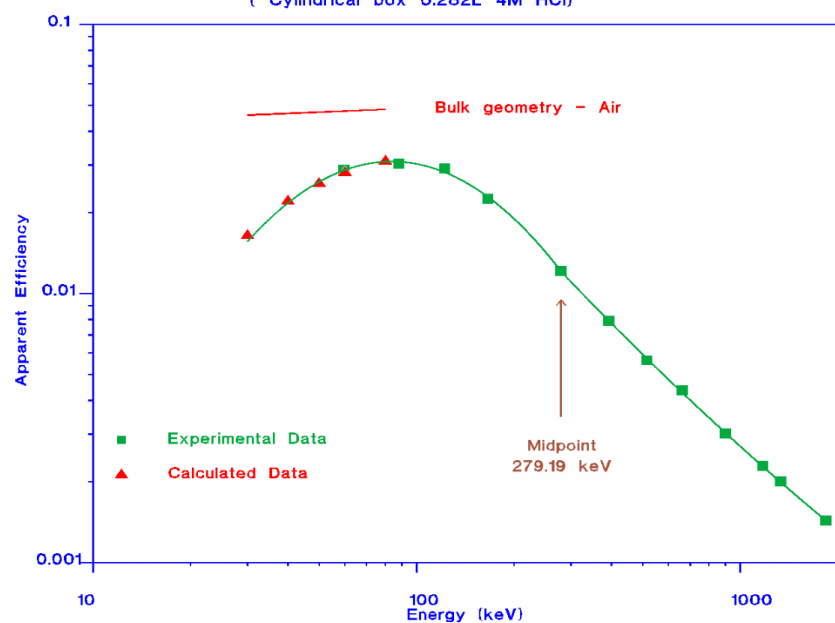


Figure 56

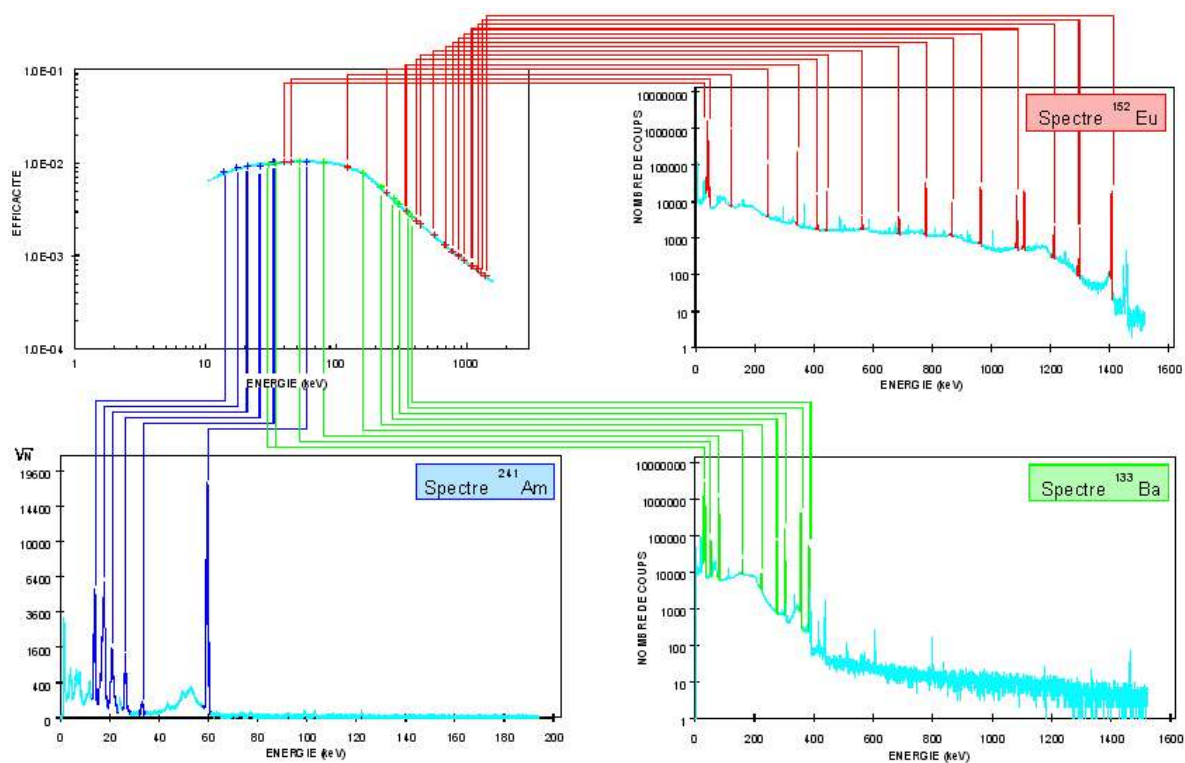


Figure 57 Efficiency curve using different radioactive sources

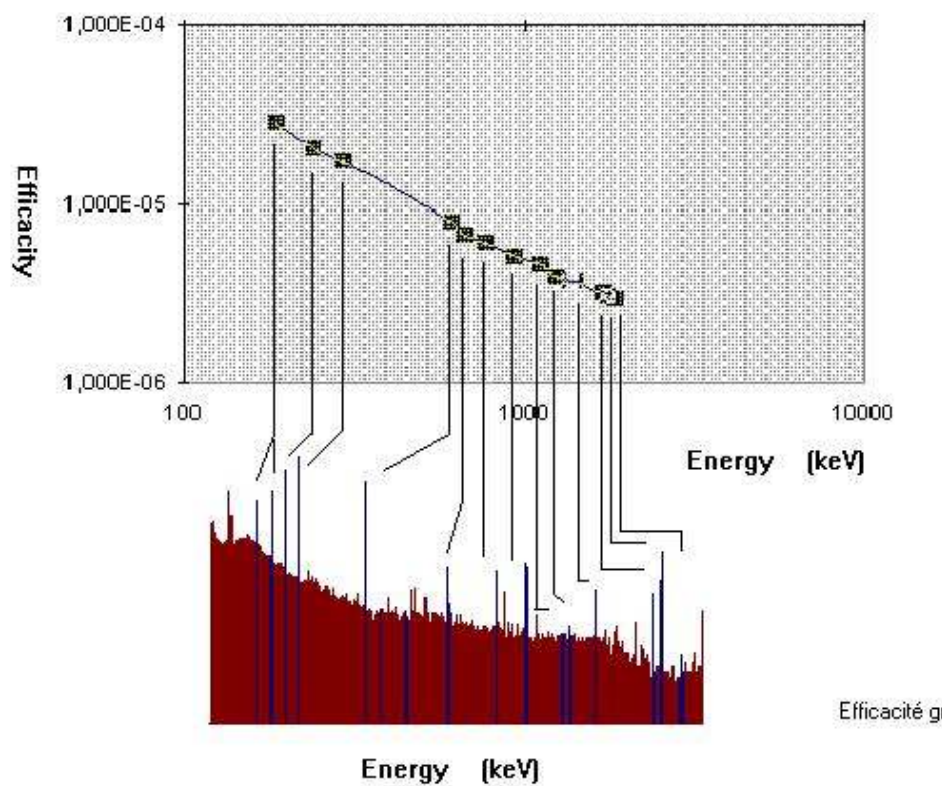


Figure 58 Efficiency Calibration using a ^{226}Ra source

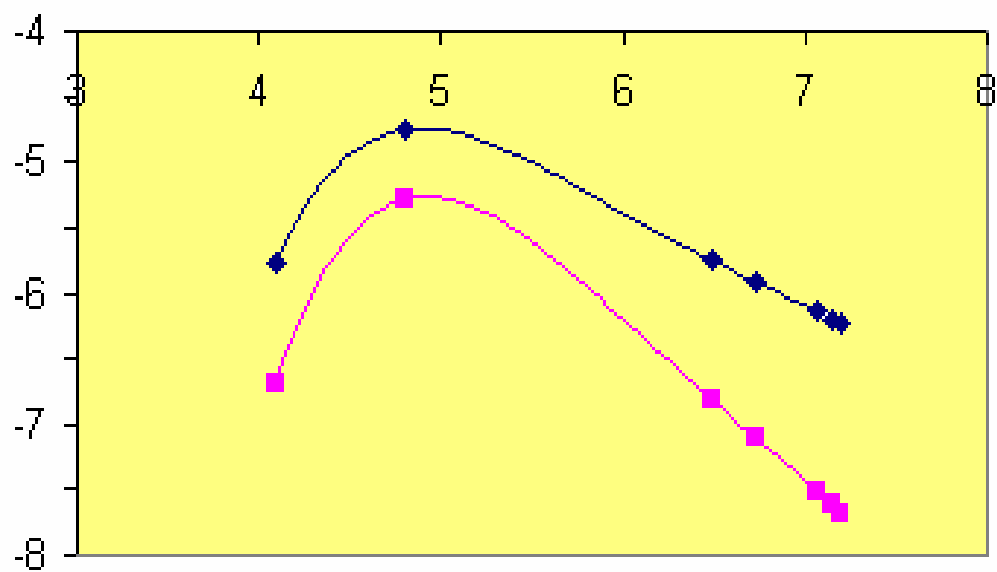


Figure 59 Comparison of Efficiency curves for two different size detectors

6.3. ANALYSIS OF THE PULSE HEIGHT SPECTRUM

The result of the measurements by the analyst is: (i) a set of typically 8192 numbers that represent the pulse height spectrum; and (ii) a few numbers that describe the energy and efficiency calibration of the detector. This section describes how these data are processed to provide information on the identity and activity of all the nuclides that are present in the sample that was counted. This data processing consists of a number of steps that are carried out in a fixed sequence.

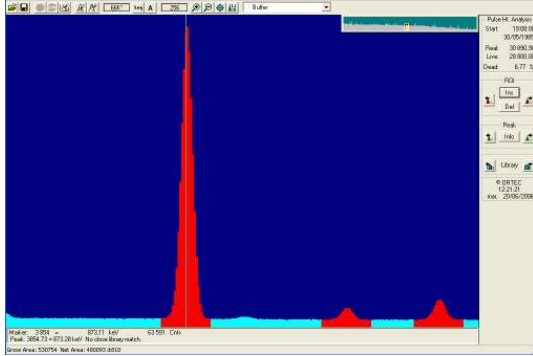


Figure 60

6.3.1. Peak Search and Deconvolution

Calculating the location of peak centroid positions allows automatic analysis of unknown spectra. The automatic peak search capability is commonly used with gamma detectors.

It is relatively easy to look at the pulse height spectrum as displayed by the MCA, and indicate where all the peaks are. It is, however, extremely difficult to translate this visual recognition process into a logical "procedure" that can be carried out by a computer.

Note that not all peaks that are present in the spectrum are useful.

The **nuclide library** is essential in this process. It is a list (inventory) of nuclides which are selected by the analyst in advance, and which contains at least the following information:

- For every nuclide listed: The isotope symbol (e.g. ^{60}Co) and the half-life.
- For every photon-line emitted by this nuclide: The accepted photon energy (keV) and the yield of that line (number of photons per disintegration).

The nuclide search is performed within an **energy window** as specified by the user. If a window of 1,00 keV is selected for example, the software will assign all the entries in the nuclide library that are between the limits of 603,94 to 605,94 keV to the first line, between 1083,94 to 1085,94 keV to the second line, etc. The example might then be:

ROI			Peak (keV)	Base	Peak		Nuclides			
#	From	To			Counts	Error	Code	keV	Y	
01	1004	1113	604,94	3171	2869	161	¹⁹² Ir ¹³⁴ Cs ¹⁹⁰ Ir	604,4 604,6 605,3	0,089 0,976 0,381	
02	1797	1821	1084,94 1086,12	2418 2381	4716 6263	288 324	¹⁴⁰ La ¹⁵² Eu ¹⁴⁰ La ¹⁵² Eu ¹²⁵ Sn	1085,2 1085,8 1085,2 1085,8 1087,1	PP 0,102 PP 0,102 0,011	
03	1846	1858	1111,89	475	5831	291	¹⁵² Eu	1112,0	0,135	

An important issue in the nuclide search procedure is the optimum size of the energy window to use. One would prefer to use a narrow window so that only the "correct" nuclides will be found. If the window is too narrow (say 0,1 keV), however, the correct nuclides will be missed if the energy calibration of the system has drifted by more than this margin (say by 0,2 keV).

The analyst will normally not know what the current energy drift is, and is therefore not able to select the window accordingly. The following practical guidelines might be of help:

- It is good laboratory practice to run a weekly calibration check on every spectrometer in use. The information collected for that purpose, will also give an indication of the magnitude of energy drift to expect. The analyst can then decide on a suitable window that will cover the drift, yet eliminate false nuclide identification.
- There are normally a few markers in a pulse height spectrum (e.g. the 511 keV line from b⁺ annihilation and the ⁴⁰K line at 1461 keV for low background counting). If these are not found, it is a clear signal that the energy calibration has drifted too much, and that a wider energy window is required.

6.3.2. Finding regions of interest

The first step is to identify groups of channels where there are probably peaks present, so that these "regions of interest" (ROI) can be investigated in detail. A number of criteria are applied one after the other, and only regions that comply with all the criteria are accepted.

A common procedure is to start at the lower channels, and calculate moving values for the average of the number of counts (A) and for the standard deviation (F). This averaging is typically performed over 3 or 5 channels. The contents of the next channel is then compared with the current value of (A + 3F). If it exceeds this limit, it is accepted as the first "signal" that a peak might be ahead, and the

data are to be tested for the next criterium. If not, the comparison continues for the next channel. The next criteria could be to start from the first channel meeting the $(A + 3F)$ test, and calculate the difference between the number of counts in consecutive channels. This difference should initially be positive (as one moves up the peak), go through zero (on the top of the peak), go negative (on the upper slope of the peak), and return to zero (as one reaches the baseline). These calculations are often carried out by determining the first and second derivatives on smooth functions fitted to the data.

If the data meet all these and other criteria in the example, it is assumed that one or more significant peaks are present in that part of the spectrum, and a ROI is set accordingly. If the criteria are not met, the search continues to higher channels. The software often allows the analyst to make the selection criteria more or less stringent. This option should be applied prudently because it can become impossible to compare data that were obtained under different conditions. The manuals are sometimes vague on how parameter settings actually affect the data, and the analyst might corrupt the results without knowing it. The major obstacle in any peak search procedure, is the baseline on which all the peaks are super-imposed. Typical problems are:

- One can not assume that the baseline will always decrease slowly to the higher channel numbers: It can increase (e.g. just below the Compton edge of a strong peak) and it can decrease rapidly (e.g. just above the edge). The baseline under the ROI is estimated by extrapolation from the lower channels. If there are many peaks in the spectrum, the ROI's become "long" and so close together that it is no longer possible to define a reliable baseline.

Some software attempts to solve the baseline problem in a process of **erosion**: A smooth baseline is calculated by first subtracting all the prominent peaks, and then eliminating the remaining small features. A fraction (say 10 %) of this is then subtracted from the pulse height spectrum. The process of smoothing and subtraction is repeated until no peaks remain. Another possibility is to define in advance where peaks are expected, and then only look for these peaks in the pulse height spectrum.

6.3.3. Nuclide Identification

The nuclide identification algorithms take into account all energy lines of a nuclide entered into the analysis library with their proper branching ratios, as well as the half-life of the nuclide. For a positive identification, a nuclide must have at least one gamma energy within the user-selected energy tolerance of an observed peak in the spectrum. Furthermore, a sufficient number of the other energies of the nuclide (if there are other energies) must also have matching peaks in the spectrum. The number of peaks that must be seen is determined by the sum of the branching ratios of the peaks with matching spectrum peaks as compared to the sum of the branching ratios of the peaks without matching

spectrum peaks. Finally, the decay time of the measured spectrum must not be excessive compared to the half-life of the nuclide.

Nuclides that pass these tests with a confidence index greater than the user selected threshold will be classified as identified.

```

Interference Corrected Activity Report      2/22/00  9:46:02 AM  Page 1

*****
*****  N U C L I D E  I D E N T I F I C A T I O N  R E P O R T  *****
*****

Sample Title:          GENIE-2000 Spectrum No. 1
Nuclide Library Used:  D:\genie2k\camfiles\STDLIB.NLB

.....  IDENTIFIED NUCLIDES  .....

Nuclide    Id      Energy      Yield      Activity      Activity
Name       Confidence (keV)      (%)      (uCi/unit)    Uncertainty

K-40       0.998   1460.81*   10.67      1.81977E-001  8.15531E-003
CO-57      0.999   122.06*   85.51      2.91508E-002  1.00247E-003
           136.48*   10.60      3.33071E-002  2.82360E-003
CO-60      1.000   1173.22*  100.00      9.93055E-002  1.65456E-003
           1332.49*  100.00      9.71557E-002  1.62720E-003
SE-75      0.340    96.73      3.41
           121.11*   16.70      1.89094E-001  9.36091E-003
           136.00*   59.20      7.55523E-003  7.03221E-004
           198.60      1.45
           264.65      59.80
           279.53*   25.20      9.62423E-002  3.72937E-003
           303.91      1.32
           400.65      11.40
RE-85      0.999   513.99*   0.43      8.53421E+000  2.31906E-001

```

6.3.4. Reliability of the nuclide identification

The analyst is sometimes confronted with the demand that "all the radionuclides in the sample" must be reported (e.g. for environmental monitoring or the certification of radioactive impurities). The reality of the situation is this:

- The procedure described will meet the requirement, but the cost for a thorough study can seldom be justified.
- If the results obtained are accepted without question, there is a possibility that a radionuclide in the sample will go unnoticed if it is not covered in the library.

The following procedure is suggested for these situations:

- Run the analyses with a library that was defined as described in previously.
- Identify the strongest peak (based on "Peak" area values only) reported for this pulse height spectrum.

(c) Calculate a cut-off value (typically 0,1 % of the strongest peak) on "Peak" area values, and evaluate all the peaks that were not assigned to library nuclides against this limit:

- If no peaks are found: The reported results satisfy the demand of the client.
- If such a peak is found:
 - Identify the nuclide responsible for this peak.
 - Revise the nuclide library.
 - Run the data processing with the revised library.

6.3.5. Peak resolution

The analyst using gamma spectrometry often has to deal with situations where two adjacent peaks overlap to some extent. The situation can be improved somewhat by using detectors with very high resolution, but the problem can never be eliminated completely. In some of the applications (e.g. activation analysis) the spectra are very rich in lines, so that three and more lines might overlap. Sometimes the lines are so close together that the "valley" between the two peaks disappears, so that the analyst can no longer be sure how many peaks are present under the envelope.

The software identify overlap (i.e. multiple peaks in a single ROI) when the expected FWHM value for a particular ROI is larger than the expected value for a single peak at the same energy. There are mathematical procedures to resolve the data in this ROI into the smallest number of single peaks with the FWHM values and tailing parameters that are applicable to this energy. The techniques for doing this fall outside the scope of this course.

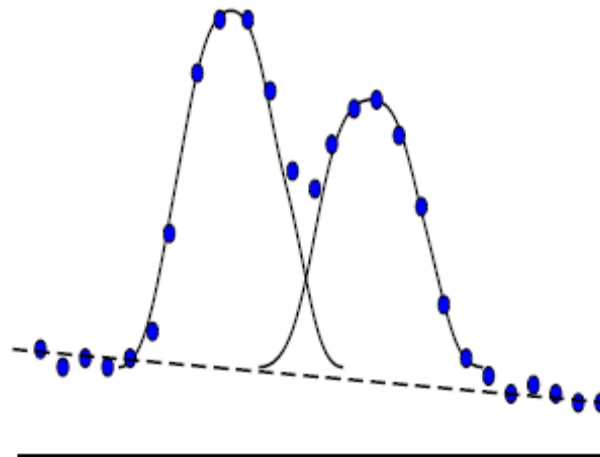


Figure 62

Once the complex combination has been resolved into a number of single peaks, their location (keV) and area values are calculated individually as was discussed

previously. There are a number of issues that the analyst must consider when applying the results presented by MCA:

- The software will normally give some indication on how fast the process converged. The smaller the number of iterations required to close the deconvolution process, the more reliable will the results be.
- All the data (i.e. number of counts in each channel) are subject to the normal statistical error. When the contribution of the first (largest) peak is subtracted from the data in the ROI, the probable error on the remaining data increases. This propagation of error continues as more peaks are "removed". The larger the number of single peaks that are necessary to describe the experimental data, the less reliable the final results become.
- It can happen that the peak search procedure fails, and the "valley" region between two overlapping peaks is assumed to be the baseline value. It is clear from the illustration that the calculated peak area values will be wrong. The chance for events like these can be reduced somewhat by prescribing a wider baseline region on the sides of the peak.

The only way to make absolutely sure that the data were not treated this way, is to visually inspect the pulse height spectrum, and ascertain how the peaks were resolved and where the baseline was placed. This approach can, for reasons of cost, not be applied to every spectrum that is encountered by the analyst.

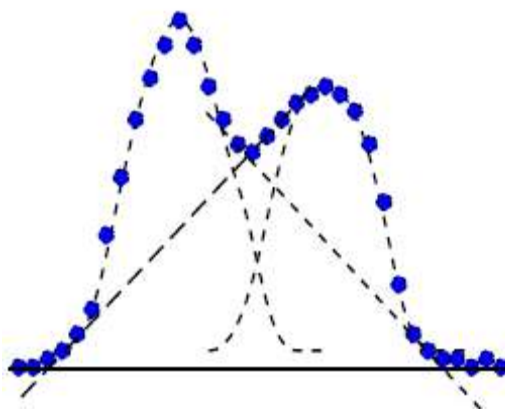


Figure 63

6.3.6. Peak area determination

The counts in the peak are calculated by subtracting a straight line or step background drawn between the end points of the region of interest set about the peak (by the peak search routine or manually). End point averaging over a number of channels is provided to reduce the error when background

determination is uncertain. The error in the net area, including uncertainty in background, is also calculated.

The data obtained can give only approximate values for the position of the peak (channels), the baseline on which it is super-imposed, its height, and its lower and upper boundaries. These are processed as described below.

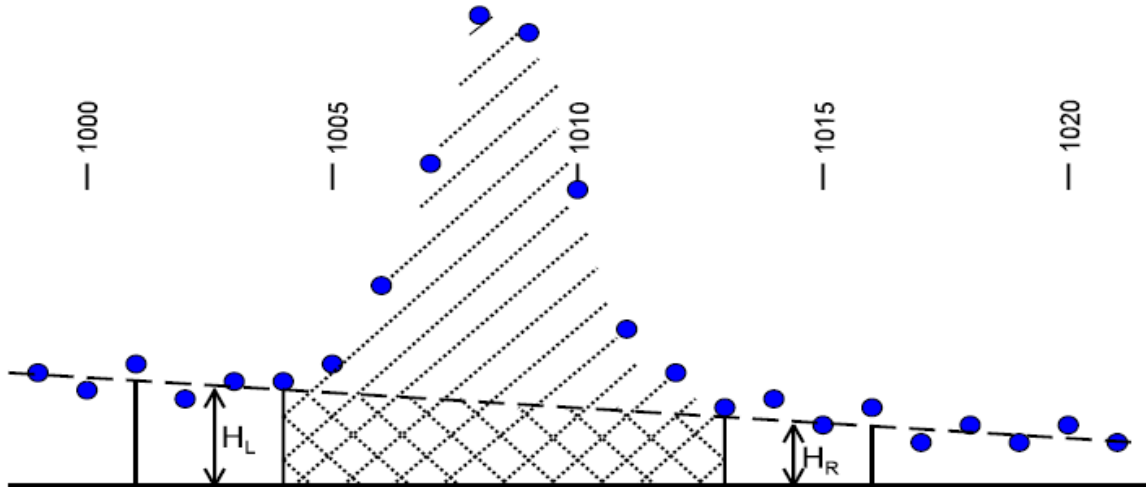


Figure 61

The FWHM of the suspected peak is calculated, and compared with the value expected for this detector at this energy. If the values agree, it is assumed that only one photon is responsible for this peak, and the peak area is determined using the equations below. If the peak is wider than expected, it is assumed that it is a combination of two or more peaks, and the more complex peak deconvolution process is applied. (*A peak that is too narrow, is normally rejected in the search process*). The peak area (number of counts) corresponding to the full energy event is calculated from:

$$\text{Peak} = \text{Integral} - \text{Base}$$

where:

$$\text{Integral} = N_{1004} + N_{1005} + \dots + N_{1016} \quad \text{Sum over 10 channels}$$

and:

$$\text{Base} = \frac{H_L + H_R}{2} \times 10 \quad \text{Peak covers 10 channels}$$

with the baseline value calculated over 4 channels on both sides of the peak:

$$H_L = \frac{N_{1001} + \dots + N_{1004}}{4} \quad \text{and} \quad H_R = \frac{N_{1013} + \dots + N_{1016}}{4}$$

The analyst can select the number of channels on both sides of the peak that will be used for calculating the "base" value. Selecting a larger number (wider basis) will improve the quality of the result by reducing the probable error, but it will cause problems when adjacent ROI's are close together.

- The software will also calculate a probable error on the nett peak area, by applying the standard counting statistics and adding the errors on the "integral" and "base" values.
- The position of the peak centroid (channel number) is also determined. This value is then converted to the corresponding energy value (keV) by using the current energy calibration parameters.
- The reliability of the peak area and position values are extremely sensitive for errors in the baseline: If the software fails in this respect, all the values calculated later become suspect.

6.3.7. Activity Calculation

The final step in nuclide analysis is to determine the intensity of the radioactivity corresponding to each isotope. The net area of the peak is directly related to the intensity, but it is also necessary to correct for the efficiency of the detector, the branching ratio of the source, and the half life (if it is desired to relate the activity to an earlier or later time). The efficiency was discussed earlier and has an energy dependence such as shown in Figure 56. The branching ratio (or yield) is used to correct the number of gamma rays observed to obtain the number of disintegrations of the source. Figure 65 shows the decay scheme for ^{88}Y and the percent of disintegrations leading to the various gamma rays.

Although the computerized spectral analysis software can perform several tasks from peak search to activity calculation, we prefer to do the calculations manually. Radionuclides can be identified by visual inspection of the photopeak and using a table of disintegration energies for the suspected nuclides to check for their existence.

The activity of the sample (Bq/kg) is calculated as follows

$$A = 1000 \cdot (N/T) / (m \epsilon P_{\gamma})$$

where N is net counts of the photopeak, T is the live time of measurements (sec), m is the mass of the sample used (g), ϵ is the counting efficiency of the specific nuclide's energy and P_{γ} is the gamma transition probability through the specific energy. No corrections for auto-absorption or other effects are made at this time.

Half-life corrections are made by multiplying the activity by an exponential factor.

$$A(\text{at time } t_0) = A e^{\frac{\ln(2) \times \text{Decay Time}}{\text{Half Life}}}$$

Where decay time and half-life must be in the same units (seconds, minutes, hours, or years).

Further specific data analysis is highly dependent upon the application, detector and electronics configuration, and is beyond the scope of this brief presentation.

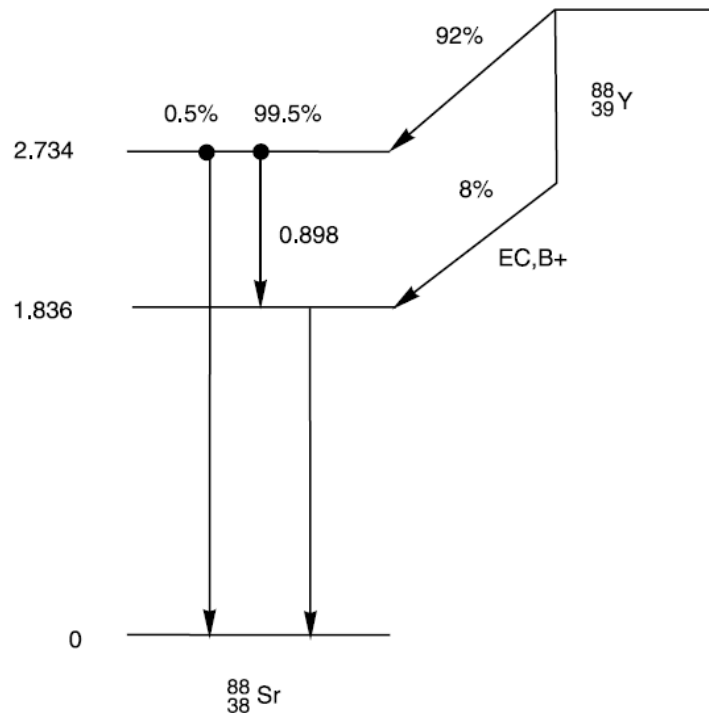


Figure 65 ^{88}Y Decay Scheme

The next phase in the process is to assign all the significant lines in the pulse height spectrum (i.e. those listed in the table) to one or more possible nuclides.

The nuclide assignment above, and the subsequent calculation of the activity values, can be performed without any logical decisions. It can be represented by the following sequence that is carried out for every entry:

- The counting efficiency at the relevant energy value is calculated using: (i) the geometry code specified by the analyst, and (ii) the efficiency calibration model and parameters that were stored previously.

$$\epsilon = f(E_\gamma)$$

This is repeated for the different photon energy values identified during the peak search phase of the procedure.

- The gross activity value at measurement (time = t) is calculated from the data in the table:

$$A_t \text{ (Bq)} = \frac{\text{Peak (counts)}}{\epsilon \cdot Y \cdot S}$$

where: **S** is the counting period (seconds).

- The gross activity value is corrected for radioactive decay to the reference date, and converted to the corresponding activity concentration (Bq.kg⁻¹ or Bq.l⁻¹) by considering the size of the sample that was counted:

$$SA_0 = \frac{A_t}{M} \cdot e^{t \cdot \ln 2 / T}$$

The probable error, based on normal counting statistics, is calculated for each of the above activity values by the standard progression of errors approach. The software normally makes provision for the analyst to select which of the calculated values are to be printed out, and what the format would be. Typical values would be:

ROI			Peak (keV)	Base	Peak		Nuclides			Activity (Bq.kg ⁻¹) corrected for decay
#	From	To			Count s	Erro r	Cod e	keV	Y	
01	1004	1113	604,94	3 171	2 869	161	¹⁹² Jr ¹³⁴ Cs ¹⁹⁰ Ir	604,4 604,6 605,3	0,089 0,976 0,381	26,9 ± 1,6 2,45 ± 0,15 6,28 ± 0,38
02	1797	1821	1084,94	2 418	4 716	288	¹⁴⁰ La ¹⁵² Eu	1085,2 1085,8	PP 0,102	- 51,4 ± 3,6
			1086,12	2 381	6 263	324	¹⁴⁰ La ¹⁵² Eu ¹²⁵ Sn	1085,2 1085,8 1087,1	PP 0,102 0,011	- 63,2 ± 3,5 633 ± 38
03	1846	1858	1111,89	475	5 831	291	¹⁵² Eu	1112,0	0,135	49,1 ± 2,9

Table 5

The software can do all these calculations very efficiently, and will present the analyst with all the possibilities and the corrected specific activity values. But it can not decide which nuclide to assign to a particular line in the pulse height spectrum. The final decision is based on the professional judgement of the analyst, and takes the following into the consideration:

- The type of sample and its origin: One would not expect the neutron deficient isotopes in samples from activation analysis. And one would assign a line in an environmental sample to the decay products of uranium and thorium; rather than to man-made nuclides.
- The presence of other confirming lines from the same nuclide; especially the stronger ones: One would rather not assign a line to a particular nuclide, if the other lines of that nuclide with larger yields are absent.
- Half-life of the nuclide relative to the decay period: If the half-life is short (say $T < 5t$), it is very unlikely that the nuclide will still be present.

The probable error reported for the final activity concentration value, is supposed to cover all the sources of error that are encountered in the process described above. It typically includes the contributions from the following:

- Statistical errors on the "Integral" and "Base" values that were used for calculating a "Peak" value for the sample that was analysed. Similar statistical errors made during counting of the efficiency calibration standard.
- Errors due to the extrapolation of the efficiency data to energy values beyond the region covered by the calibration standard(s). This contribution often is so large that the reported value becomes useless.
- Errors due to uncertainty in the half-life value used for decay corrections. The precision of these values normally would not allow corrections for more than about 4 to 5 half-lives.

6.3.11. Qualitative analysis of unknown samples

The nuclide library is crucial for gamma spectrometry because a quantitative measurement can only be performed using a library that is suitable for that application. This presents a problem for a sample that contains unknown nuclides: such a library does not exist yet. The analyst is therefore forced first to identify the radionuclides in the sample, and then design a suitable library accordingly.

This process requires considerable patience and professional judgement. The following guidelines should be considered:

- The analyst can only work with the pulse height spectrum from the gamma spectrometer; and nuclides that do not emit hard gamma rays (e.g. ^3H , ^{90}Sr and β^- -emitters), will not be detected. The results can therefore be misleading.

- It is better to start with the stronger lines at the high energy end of the spectrum (where there are fewer lines), and then work "backwards" to lower energies and weaker lines.
- Once a nuclide has been identified: (i) use its other lines to confirm this identification, and (ii) also assign these other lines to this nuclide.
- Calculate the differences between the position of the peak (keV) and the true energy of the associated photon. Use all the available data to estimate the energy bias of the instrument due to the drift.
- Use whatever other information that is available to exclude or confirm the presence of a nuclide: (i) origin and history of the sample; (ii) half-life of a nuclide relative to the decay period of the sample; and (iii) radioactive precursors or decay products of a nuclide.
- Be aware of the odd properties of gamma spectrometry: escape peaks (often from high energy lines outside the region covered), and sum-coincidences at high efficiency or high counting rates.
- Accept that: (i) this is an iterative process that must be repeated a number of times, and (ii) a number of weak lines will remain unidentified.

6.3.8. Defining a new nuclide library

Setting up a nuclide library for a particular application, is extremely important. The time and effort spent in the careful design and documentation of a library, is more than justified by the confidence with which it can be applied in future. The process always involves a compromise:

- One would like to include many nuclides and lines to make sure that all the possibilities are covered, and that no nuclide will escape the search.
- One would like to include only a few nuclides and the minimum lines, to speed up the data processing, and to eliminate the need for working through reams of paper with false "hits" searching for the best option.

This design process can, however, not be described in a number of fixed instructions that will guarantee the desired product. The analyst should, however, keep to the following guidelines:

- (a) Formulate a series of criteria for the particular application, on which nuclides should be **included** and **excluded**. The following are examples:
 - Sample irradiated in the reactor and counted after about 24 hours for activation analysis: only neutron-rich nuclides with $T > 6$ hours.
 - Fission Mo 10 99 weeks after production: only fission products with $T > 1$ week.
- (b) Use information on these nuclides only from a single data source used by all the personnel in the group (to keep all the information internally consistent).

Eliminate all the weak lines, retaining not more than four of the strongest lines in the energy region covered by the detector.

- (c) Adjust the (shorter) half-life of a decay product to the (longer) value of the precursor where applicable; eg ^{140}La (1,7 d) and ^{140}Ba (12,8 d).
- (d) Document the criteria used, the nuclides selected and the lines eliminated so that: (i) all the other users of this library can be kept informed; and (ii) questions can be handled in the future. Make provision for the orderly (and documented !) revision of the library as small problems are identified and eliminated during its use.
- (e) A new library should initially only be used by the person who designed it. Once it has been properly debugged it can be released for general use.

6.3.9. Peaks not found in the spectrum

Data processing software normally makes provision for calculating the minimum detectable activity (MDA) values for nuclides. It is either done for every nuclide specified in the library, or only for those nuclides that did not give significant peaks. The process typically covers the following action:

- The energy of the primary line of the nuclide (normally the strongest line without interference) is used to calculate and specify the channel number where the corresponding full energy peak is expected to be.
- The width of the expected peak (number of channels) at that position is calculated, and the contents of all the channels over this region are integrated. This integral is equivalent to the value expected for the "Base" if a peak should have been identified.

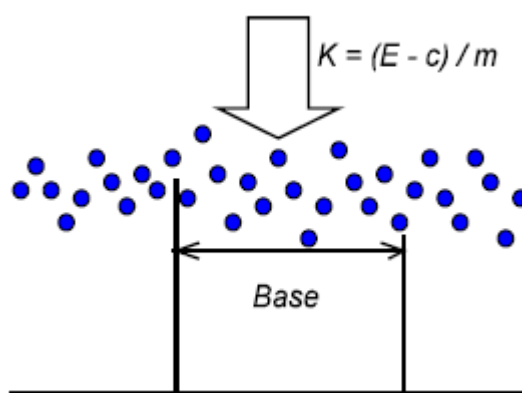


Figure 66

The detection limit (DL) or the smallest significant "Peak" value (counts) is calculated by applying an equation that is similar to the one used in gross counting techniques:

$$DL = 2.71 + 4.76 \cdot B^{1/2}$$

where the "background" reading (B) is replaced for this application by the "Base" value.

- This "minimum detectable peak area" value is converted to the corresponding MDA value (Bq.kg^{-1} or Bq.l^{-1}) as discussed previously for significant peaks.

6.3.10. Background corrections

In gamma spectrometry background contributions can be identified (e.g. 40K at 1461 keV and cosmic rays at 511 keV) and they normally do not affect the analytical line. Background corrections are therefore not required.

Note: The full-energy peak in the pulse height spectrum is super-imposed on a relatively smooth basis. The value for this "base" that is subtracted from the "integral", is sometimes referred to as "background". This practice is misleading, and should be discouraged; rather use "base" or "underground". There are isolated cases where the analyte line is also present in the pulse height spectrum obtained without a sample in the counting vault (e.g. ^{214}Bi at 609 keV). Under these circumstances it becomes necessary to measure a pulse height spectrum for background separately, and calculate the effective contribution at the analyte line. This blank value is then subtracted from the measured activity concentration bearing the following in mind:

- The background spectrum must be processed repeatedly using the "mass" values that apply to the corresponding samples.
- The interference is often caused by the sample container, and not by the surroundings.
- The calculated background activity is subtracted, and not the background spectrum itself.

6.3.11. Distortions of the pulse train due to pile-up

Relatively long pulse decay times are used in the initial stages of the hardware to reduce distortions due to electronic noise. At high counting rates there is then a possibility that a second event in the detector might occur before the pulse from the previous event has decayed. The second pulse will "ride" on the tail of the first one.

This effect is known as **pulse pile-up**, and will result in a measured pulse height value for the second pulse that is higher than the true value. The end result of pile-up in the pulse train is that some of the pulses in the full-energy peak will be shifted to higher values; giving a large high-energy tailing of the peak in the pulse height spectrum.

The effect of pulse pile-up can be eliminated by the hardware, using the following procedure:

- A voltage sensor is used to continuously monitor the input of the ADC.
- If the voltage is close to zero: A new pulse is accepted and processed by the ADC.
- If the voltage is not close to zero: The ADC is closed, and will not accept any new pulses until a value close to zero is reached. (*Eliminating distorted pulse creates another problem: some of the incoming pulses are rejected and can not be processed.*)

6.3.12. Dead-time losses

ADC requires some time to process an incoming pulse. During this period, known as the **dead time** of the instrument, it can not accept another pulse. This second pulse is therefore lost. This problem is aggravated by the pile-up rejection discussed in the previous paragraph

6.3.13. Time measurement and corrections

It has been shown that some of the pulses are lost due to pile-up rejection and the inherent dead time of the ADC. This will lead to inaccurate results. The hardware can compensate for these losses.

Another problem arises for samples containing short-lived nuclides, if the counting period is of the same order as the half-life of these nuclides, and the dead time decreases during the counting period. The system compensates for lost pulses by adding more time for counting at the **end** of the selected period. But the activity of the short-lived nuclide is then less than the value at the **start** of the measuring period, when more pulses were lost due to a higher dead time of the ADC. The net effect is that the dead time correction under-compensates for the short-lived nuclides; and over-compensates for the long-lived ones.

6.3.14. Minimum detectable activity (MDA)

The calculation of Minimum Detectable Activity for a given nuclide, at the 95% confidence level, is usually based on Currie's derivation (Currie, L.A. (1968) Anal. Chem. 40:586.), with one simplified formulation being:

$$\text{MDA(Bq/unit wt)} = \frac{2.71 + 4.66(\sigma)}{T \cdot \text{EFF} \cdot Y \cdot \text{wt}}$$

where

σ is the standard deviation of the background collected during time T over the energy range of interest

T is the collect time (sec)
EFF is the efficiency at the energy of interest
Y is the Branching Ratio
wt is sample weight

This formulation takes into account both kinds of errors – false positive and false negative, and yields the smallest level of activity which can be detected with 95% confidence, while also having 95% confidence that “activity” is not detected falsely from a null sample. When the measurement is made on a ‘blank’, with no activity, but with the same form and density as an actual sample, the calculated MDA is an a priori estimate of the best sensitivity that can be expected from sample measurements. When the calculation is applied to a spectrum collected from an actual sample, the background at the energy of interest will in most cases be higher, due to interference and Compton scattering from other nuclides in the sample. Thus, the MDA for an actual sample, computed a posteriori, will be somewhat higher than the a priori estimate.

The MDA – also referred to as Lower Limit of Detection (LLD) – can be improved by increasing the efficiency of detection, decreasing the background, or, for a given experimental setup, by increasing the collect time or the sample size. It is frequently necessary to select the appropriate collect time to ensure that the measured MDA will be below the action level mandated by the count-room procedures.

7. SPECIAL TOPICS

7.1. Low level gamma ray counting – Geometry

Large volume **HPGe** detectors have become dominant over other detector types for low level gamma ray spectroscopy because of their inherently good **resolution** and **linearity**. Since the majority of all gamma spectroscopy applications require the analysis of more complex, multi-radionuclide samples, the following discussion will be limited to the application of HPGe detectors to low level counting.

The sensitivity of a HPGe spectrometer system depends on several factors, including detector **efficiency**, detector **resolution**, **background radiation**, sample constituency, sample geometry and counting time. The following paragraphs discuss the role these factors play in low level gamma ray counting.

Efficiency: For the scientists who are counting weak samples with lower gamma energies, for instance 100-800 keV, the following subtle considerations to the detector design are important to system performance:

- a. The detector should have an adequate diameter. This assures that the efficiency at medium and low energies will be high relative to the efficiency at 1.33 MeV, where it is bought and paid for.
- b. The detector-to-end-cap distance should be minimal – five millimeters or less. The inverse square law is real and will affect sensitivity.
- c. The detector should be of **closed end coaxial** geometry, to assure that the entire front face is active.

Resolution: Generally, the superior resolution of a HPGe detector is sufficient enough to avoid the problem of peak convolution, (i.e., all peaks are separate and distinct). The sensitivity of a system improves as the resolution improves because higher resolution means that spectral line widths are smaller, and fewer background counts are therefore involved in calculating peak integrals.

Since the sensitivity is inversely related to the square root of the background, that is, improvements in resolution will not improve sensitivity as dramatically as increased efficiency.

$$Sensitivity = \frac{1}{\sqrt{Bkg}}$$

Background radiation and sample constituency: Interfering background in gamma spectra originates either from within the sample being counted (**Compton**-produced) or from the environment. If the sample being analyzed has a high content of high-energy gamma emitting radioisotopes, the **Compton**-produced background will easily outweigh the environmental **background**. For extremely

weak samples, the environmental background becomes more significant. Obviously, massive [shielding](#) will do little to improve system sensitivity for low energy gamma rays in the presence of relatively intense higher energy radiation. However, Compton-suppression can be very effective in reducing this background.

[Sample geometry](#): An often overlooked aspect of HPGe detector sensitivity is the sample geometry. For a given sample size (and the sample size should be as large as practicable for maximum sensitivity), the sample should be distributed so as to minimize the distance between the sample volume and the detector itself.

This rules out analyzing “test tube” samples with non-well type detectors, or “large area flat samples” with standard detectors. It does rule in favor of using re-entrant or [Marinelli-beaker](#)-type sample containers, which distribute part of the sample around the circumference of the detector.

Another aspect to consider is the absorption of γ -rays inside the sample material itself, and the influence of the dimensions of the sample container on this. For water and other light material (eg coal or vegetation) the absorption of hard γ -rays (above 300 keV) in a layer of the material less than 20 mm thick is negligible. For more dense material (eg metals and heavy minerals) and for lower energy γ -rays, this selfabsorption can no longer be ignored. One can, however, compensate for this by: (i) using thinner samples, or (ii) making corrections for absorption. It is generally better to use a large but thin sample directly on the face of the detector to obtain the maximum counting efficiency; a full Polytop container lying flat (diameter about 25 mm) will cause less selfabsorption than one standing upright (height about 60 mm).

Another approach that can be used for very large liquid or powder samples (more than about 500 mℓ), is to pour the material into special containers, i.e. Marinelli beakers, that fit around the detector head. In this arrangement the maximum amount of material is brought as close as is possible to the sensitive volume of the detector.



7.2. High count rate gamma ray systems

High count rate applications require special techniques to assure good [resolution](#) and/or good [throughput](#). In general, “high count rate” is used to refer to incoming count rate, that is, the number of events seen by the detector. The term “[throughput rate](#)” may be of more interest to the researcher, being a measure of the rate at which the system can accurately process these incoming counts. In high count rate HPGe detector applications, problems such as the loss of resolution, excessively long counting times, erroneous peak to background ratios, inaccurate counting statistics or system shutdown due to overload and saturation begin to appear. In some experiments, the solution to these problems merely lies in reducing the incoming count rate to the detector, or by employing electronics which inhibit the processing of pulses through the electronics when events are occurring so fast that they are overlapping (pulse pileup). In this latter solution, system throughput will of course be reduced, but parameters such as [resolution](#) will be enhanced. Note that the component’s performance is not only affected by the incoming count rate, but by the relative energy (amplitude) of the incoming counts as well. Each element in the chain can be optimized for high count rate performance.

For the detector itself, the [charge collection time](#) is the limiting factor, and this parameter is a function of the detector geometry – when a photon interaction takes place, charge carriers in the form of holes and electrons are produced, and the time taken for these carriers to be swept to the p and n electrodes of the detector is the time for full energy collection. In a germanium detector, this time is a function of detector size, as the charge carriers travel about 0.1 mm/ns. As the charge collection time increases, the Amplifier must take a longer time to process the signal and develop its linear pulse, or else not all of the incident energy will be reflected in that pulse (“[ballistic deficit](#)”). Thus, larger detectors require longer [amplifier time constants](#), or more sophisticated peak shapes.

Some ways to address high count rate in the detector include moving the detector farther away from the source, or collimating the detector – which in both cases reduces the number of events seen by the detector – or using a detector of lesser efficiency. The detector in the latter case will ‘see’ fewer events, and furthermore will have a lower charge collection time.

7.3. In situ gamma spectroscopy

In situ measurements will rarely be the complete solution, but for many cases it can greatly reduce the number and cost of gross [dose rate/countrate] field measurements, and number of samples for laboratory measurements. Typical Applications include:

Decontamination Assessment

Determination of near-surface ground contamination.

- Determination of subsurface contamination by “well logging”.
- Building Contamination Assessment
 - Wall, floor, and/or ceiling activity measurements. Pipe and duct holdup measurements.
 - Assessment of status of decontamination efforts.
- Radioactive Waste Measurements
 - Measurement of nuclides and activity of boxes, bags, drums, and other objects.
 - Free release measurements.
- Environmental Monitoring
 - Determination of site natural background. Deposition following real or suspected accidents. Field assay of air particulate and iodine cartridges.
- Public Health Measurements
 - Immediate results from the measurement of suspected contaminated areas.
- Health Physics Measurements
 - Total room contamination [walls, ceilings, non-removable fixtures].
 - Determination of the cause for abnormal gross/dose rate survey indications. Free-release determination of objects.
- Nuclear Facility Maintenance
 - Identify nuclides in pipes or tanks *without* opening them. Quantify hard to sample residual activity from plateout.
- Emergency Response Teams
 - Immediate answers to questions like “What and how much has spilled?”, “What set off the gross counter alarm?”, and “Is that wound contaminated?”

The following documents were used in this compilation:

Nuclear Chemistry, Document Reference: NWU-NC-TH-05, Theme 5,
Measurement of Radioactivity, Part 3: g-Spectrometry, Original document
compiled by: Mr Barnard Smit, Adapted for use NWU by: Prof Dr Arnaud
Faanhof, March 2006. NECSA, South Africa.
University of Texas at Austin, Nuclear Teaching, Sheldon Landsberger.
Practical Gamma-Ray Spectrometry by Gordon Gilmore and John Hemingway,
John Wiley & Sons, NY, 1995.
CEA Documents
Canberra web site
NY University web site