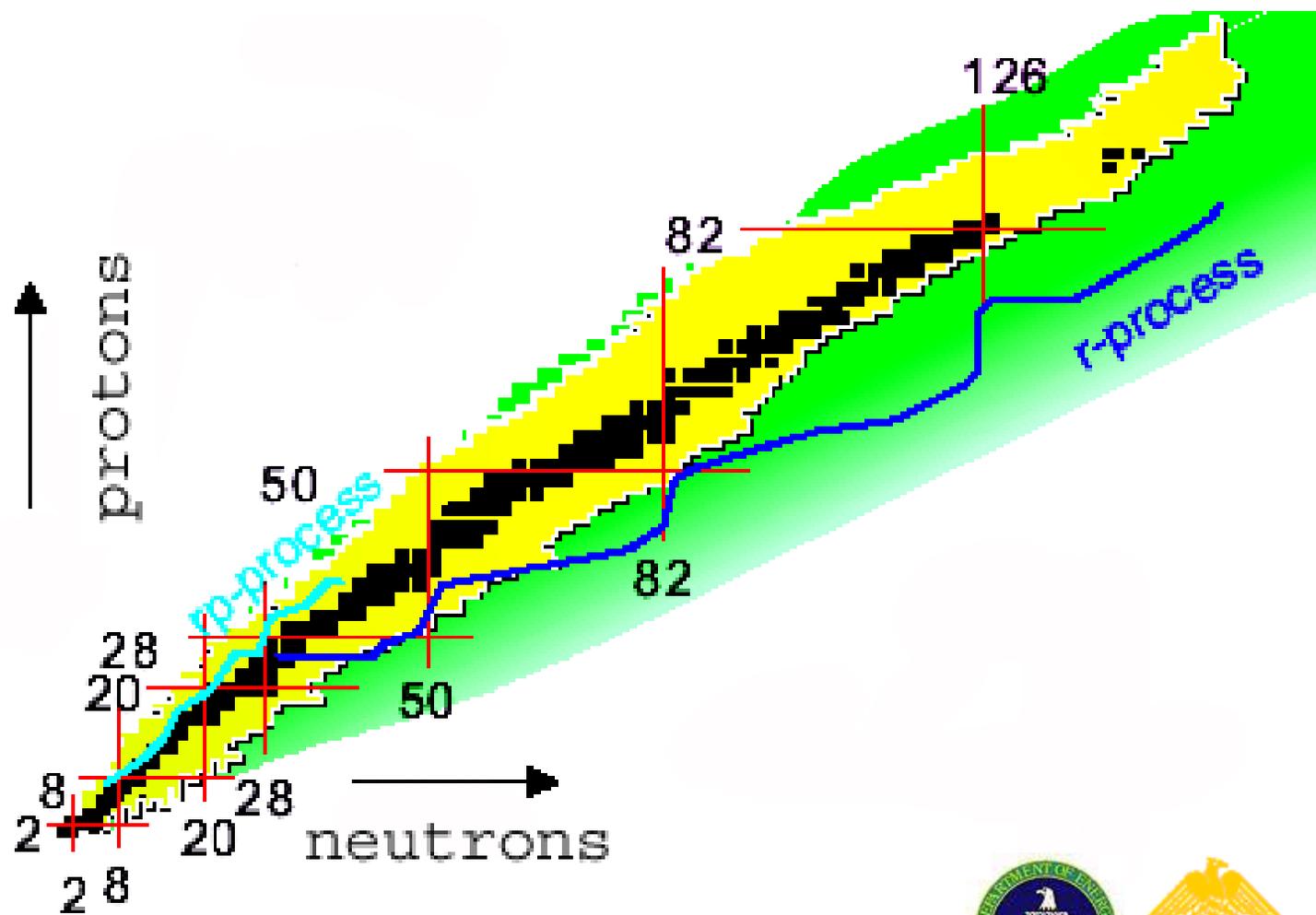


CHE-362 Laboratory Manual

Nuclear and Radiochemistry Summer School 2013



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INTRODUCTION

Welcome to CHE-362, Nuclear and Radiochemistry Laboratory. The laboratory and lecture courses are given in parallel. While they are complementary, the order of topics in one does not dovetail well with the order of topics in the other. Thus, it is especially important that you acquire the necessary background information for each experiment beforehand by reading the assigned pages in this manual. There will also be pre-lab sessions to promote understanding of the experiments and highlight safety aspects. This course is designed to (1) acquaint you with the types of instrumentation and techniques commonly used in radiochemistry and nuclear chemistry laboratories; and (2) develop your scientific writing skills.

Grading Policies

Your grade is based on your laboratory notebook (20%), pre-lab quizzes (20%), laboratory reports (60%). There is no final exam in this course.

The laboratory notebook grade is based on how well the notebook is kept according to the “Laboratory Notebook Guidelines” described below. **Each lab describes essential elements that should be in your notebook and final report.**

The pre-lab quizzes are designed to test student knowledge of the background material and will be based on material from the written lab module. They may be multiple choice or short answer question and should take no more than 10 minutes to complete.

This year we require that you hand in individual laboratory reports for 6 of the 9 experiments assigned—it will be your choice as to which reports you wish to write up. Take note of report deadlines and manage your time appropriately. Consult the guidelines found later in this manual for assistance on how to write these reports. Each experiment will contain specific guidance on what information should be included in the report. Reports are due at 9:00 AM on their due dates. They are given to the Teaching Assistants in person or they are placed in the Teaching Assistants’ mailboxes in B. 801. They may be handed in up to 24 hours late with a 20% reduction in points. Reports are graded and handed back in a timely fashion.

Schedule

The course schedule is found on the web at <http://www.bnl.gov/ncss>. Before you perform any of the experiments described in this manual, you will complete mandatory training in general laboratory and radiation safety.

Students will be randomly assigned to working teams of three for the duration of the course. Pre-lab lectures begin promptly at 1:00 in Room 31 Lecture Room in 801.

These lectures typically last 30-45 minutes depending upon the complexity of the experiment. Laboratory experiments are performed in Rooms 32/33 (Chemistry Laboratory) and/or Room 40 (Counting Laboratory), both rooms being upstairs in B. 801. Teaching Assistants and/or the Course Instructor will be available for assistance during the entire lab session.

Remember to save all your data on your memory sticks so that you are not scrabbling at the last minute. Also note that many labs involve compilation of data from your classmates. TA's will ensure that this aspect remains organized facilitating final report write-up.

LABORATORY NOTEBOOK GUIDELINES

The laboratory notebook is the most important component of good laboratory performance since it is a permanent document of laboratory work. Therefore, a notebook will be supplied to you before the start of this course. It is a bound journal with numbered pages. Treat it as an accurate and unambiguous record of your laboratory work. The grade in this course will be a direct reflection of the content of your notebook. Your notebook is a primary record. Write legibly. Do not reproduce material from other sources such as your Laboratory Manual without appropriate acknowledgment. It is important that all data be recorded in "real time," i.e., enter measurements and perform calculations as they are done. Waiting to do calculations after the lab is over is a major source of error in this course and will certainly have a detrimental effect upon your course performance. When your notebook is collected for grading, the graders will be looking for enough explanatory information that someone else with a similar background could, from your notebook alone, repeat your work.

The following guidelines will be helpful:

Notebook Basics

Record all data in permanent ink. Test your ink in the back of the notebook for water solubility. No pencils will be allowed in the laboratory.

Reserve the first few pages of the notebook for an index or table of contents. On the front inside cover write:

- your name
- phone number
- e-mail address
- semester date
- laboratory course
- instructor's name

Notebook Format

The following format can serve as a template for keeping an accurate and useful laboratory notebook:

Title: — The start of each notebook page should include the title of the experiment. The first page of the experiment should also include a reference to the relevant pages in the laboratory manual.

Introduction: — The introduction shall state the purpose of the experiment (what is the scientific principle or theory), what is going to be measured

Procedure: — There is no need to explicitly copy a given procedure from your lab manual. A brief outline of the steps to be followed is all that is necessary. If you are assembling a detector system or wet chemistry system, a rough sketch of your apparatus is appropriate. Staple or tape any other relevant handouts, procedure modifications or report forms to your laboratory manual or to the notebook.

Observations: — Identify all observations and numeric results in a clear manner. Be sure to include succinct observations of any experiment details that you observed. Remember, the notebook is a journal of your experiences in the laboratory.

Data: — Include all raw data in tabular form. Many of the lab modules already have structured tables for you to fill in. These can be taped into your notebook for later use. Also, include a sample calculation for any numeric results with units clearly indicated. Include statistical measures and true values where appropriate.

Using the Notebook

No data of any type is to be recorded on scrap paper. All data and observations must be written directly into your notebook and dated.

Use a table format for data whenever possible (often structured data tables will be provided in the lab for you to fill in). This makes comparison of results and post-hoc data analysis much easier. The column headings of tables should show units and symbols. The table should have a title and sequential number for reference purposes.

Mistakes or errors should be crossed out in a way such that they are still legible. Never erase or obliterate data or tear pages from the notebook. If pages are to be skipped, cross-out those pages but leave the numbering sequence intact.

The usual rules regarding significant figures should be observed. Never report more significant figures than warranted by the measurement (however, when performing calculations, it is wise to record a reasonable number of excess digits since premature truncation of results may result in loss of accuracy.) The final result should contain the number of significant digits required by the least accurately known lab measurement.

In general, the left hand page of the notebook is reserved for calculations and notes. Printouts from spreadsheets, copies of graphs and spectra should be permanently attached to the notebook. Sometimes it is helpful to print outputs at a reduced size for this purpose. These attachments should be in sequence with the experimental details and should be permanently bound to the notebook. Tape or a glue stick work quite well for this purpose. Be sure to keep a record of the filename of any data

stored on a computer or disk.

A rough sketch or representation of spectral data, chromatograms, titration curves, *etc.* should be recorded in the notebook. Be sure to label the axes with measurement units and give a title to the drawing. A reference of the file name for computer data should be recorded if the data is stored on a disk or server.

If any unusual experimental equipment is used, sketch the setup or include a reference to the directions for setting up the apparatus.

Cite the source of any chemicals that are used. A manufacturer's name, grade of chemical, and certified purity are important pieces of information that may not be available after leaving the lab.

Record the name and model number of any instrument used in your experiment.

SCIENTIFIC REPORT WRITING GUIDELINES

Introduction

Every laboratory experiment will require a written scientific report so in addition to giving you experience with the theory and practice of nuclear and radiochemistry, the course will also challenge your thinking skills and the ability for you to convey your conclusions **concisely** in a written format. Laboratory reports will be graded not only for technical content but also for writing and style. This guide will provide you with a standardized format you should follow when writing your reports. Additional guidance concerning what to include in your reports will be contained at the end of each laboratory module.

Resources for Further Guidance

1. <http://www.cofc.edu/~kinard/NuclearSummerSchool/NSSHomepage.htm>
(contains generalities and specifics that are applicable to this course)
2. http://www.ncsu.edu/labwrite/res/res-studntintro-lab_parts.html (an excellent resource for laboratory report writing which also gives examples).
3. <http://www.ncsu.edu/labwrite/lwr-home.html> (an excellent resource for the planning of writing laboratory reports)
4. <http://www.nuc.berkeley.edu/dept/Courses/NE-104A/How2rept.pdf>

Need for Report Writing

Science professionals spend a significant amount of time writing reports, papers, and memoranda. The quality of your writing will be one of the criteria used to judge your performance and is an essential, marketable skill. Training and practice in scientific writing is an important part of your education. Contrary to the general student academic environment, reports are not written to demonstrate the writer's competence. In the professional world, competence is expected and the report is read to answer specific questions such as what was done, why was it important, or what new knowledge was gained. Keep these goals in mind when you write your laboratory report.

Tools

All laboratory reports must be prepared using computer-based word processing software. Graphs and tables, regression analysis, and equations shall be prepared using computer based software. Computers and laser printers are available for your use in B. 801, Room 31 (Classroom) and in Room 40 (Counting Room).

Time Required

The most frequent complaint from students about these laboratory reports is the time required to write them. However, the time you spend here to develop or improve

your scientific writing skills will be invaluable to you in your future careers. The amount of time you spend to write a report is unique to each individual but plan on spending 2-3 hours to complete the work on an individual report. In order to minimize the amount of time spent it is essential that you plan the writing of your report. The following suggestions might be helpful: Analyze your data while the Course Instructor and Teaching Assistants are available for questions (i.e. don't start writing a report at midnight!). The best time to accomplish this is at the end of the laboratory period.

General Considerations

I don't expect these reports to be long narratives describing every minute aspect of your work. As a general rule, try to keep the the body of the report to within 5 pages plus any attachments for required graphs, tables or spectra. Your notebook will have a detailed source of information on your laboratory experiences including observations, raw data, sketches of sytems etc... and so it is not necessary that this report have all that same information.

Note: All work except for data analysis must be an individual effort. You may work with your laboratory team partners in analyzing data. Often it will be necessary to compile data from other teams. Your report should then reflect your individual analysis of any trends and results. The presentation of the data and the interpretation of the analysis in your report is your individual effort.

Your goal is to write as concisely and clearly as possible so that the reader can grasp the information quickly and could accurately duplicate or expand your work. Also remember that the purpose of your reports is not to prove you arrived at a correct answer.

Each laboratory experiment is set up to demonstrate some theoretical principal. You will receive the most credit when you demonstrate an understanding of that principal and when you compare your actual data obtained with theoretical predictions. You are encouraged to consult outside literature, some of which is available in B. 801, Room 31. There is a photocopier down the hall from the TA Office. Photocopy relevant information and return the material to the library. **Please do not remove the books from B. 801.**

Basic Elements of the Scientific Report:

- Introduction:
 1. Title of Experiment
 2. Your Name and Date
 3. Summary
 - Procedures
 - Results and Data Manipulations
 - Discussion
 - Conclusions
 - References
-

Introduction

This section provides a summary of the basic principles that were demonstrated by the laboratory experiment. The following may be included in the Introduction:

- Background about the analysis to be carried out.
 - Reason/s why the research was undertaken.
 - Statement of the hypothesis (an idea or concept that can be tested by experimentation) if there is one.
 - An explanation of the different techniques used and why they are used.
 - A statement of the objective/s - what you hoped to achieve.
 - What was the purpose or objective of the experiment/research?
 - Why was the experiment/research conducted in a particular manner?
 - Why was it important in a broader context?
-

Procedures

The procedure section briefly summarizes what you did in the experiment. It can be a very brief statement about how you went about the experiment to arrive at your objective(s). Leave any detail here to your notebook.

Results

This is the heart of the document where you report the results of the experiment. Break the reporting of your final results into components parts that link back to the actual procedures you followed in the lab module (and what was documented in your notebook). This section describes in narrative form how the data was manipulated and gives any equations and procedures used in this manipulation. For each manipulation (calculation), a sample should be provided. If you are doing this on a word processor you can simply leave space and handwrite in any sample calculations. The final results of the data analysis are reported in this section. **Massive quantities raw data (not refined statistically) are expected to be logged in your notebook and need not appear in the final report unless asked for.** Again, any tables or graphs are to be included in this write-up. Error analysis, if performed, is also presented here. Generally this section is a concise summary of the results but does not contain any discussion of the implications of the data (that will wait until the Discussion Section). The following should be included in your Final Results:

- Tables of data and graphs when ask for.
- Brief statements of the results in the text (without repeating the data in the graphs and tables). When writing about each picture, graph or table, refer to it parenthetically e.g. (Figure 1).
- Sample calculations & sources of error

It is not necessary to show each and every calculation you do. Include a neat and clear example of each unique kind of calculation. Start with definitions, show substitutions, and simplifications leading to an answer.

example: To find the area (A), the formula used was
 $A = \pi (r)^2$ where (r) = radius. For the first circle,
 $A = \pi (4 \text{ cm})^2$
 $A = 50 \text{ cm}^2$.

List the type of error and how it influenced the results.

examples: Error in measuring process: Visually comparing object lengths with the meter stick was not perfect.
Error in reaction time: The time to stop the timer may not be the same as for starting timer.

The Results section should be written in the past tense and passive voice, avoiding the use of "I" and "we".

Discussion

State your interpretation of your findings, perhaps comparing or contrasting them with the literature. Reflect on your actual data and observations (each module will have questions to answer at the end to help you along in this process). Discuss the

significance of any trends, or lack of, observed in your results. **This is where individuality should shine!** Explain or rationalize errant data or describe possible sources of error and how they may have affected the outcome. The Discussion must answer the question "What do the results mean?" It is an argument based on the results. Furthermore, answer any post-lab discussion questions that are asked. For your guidance, each lab experiment has a list of discussion questions.

Conclusion

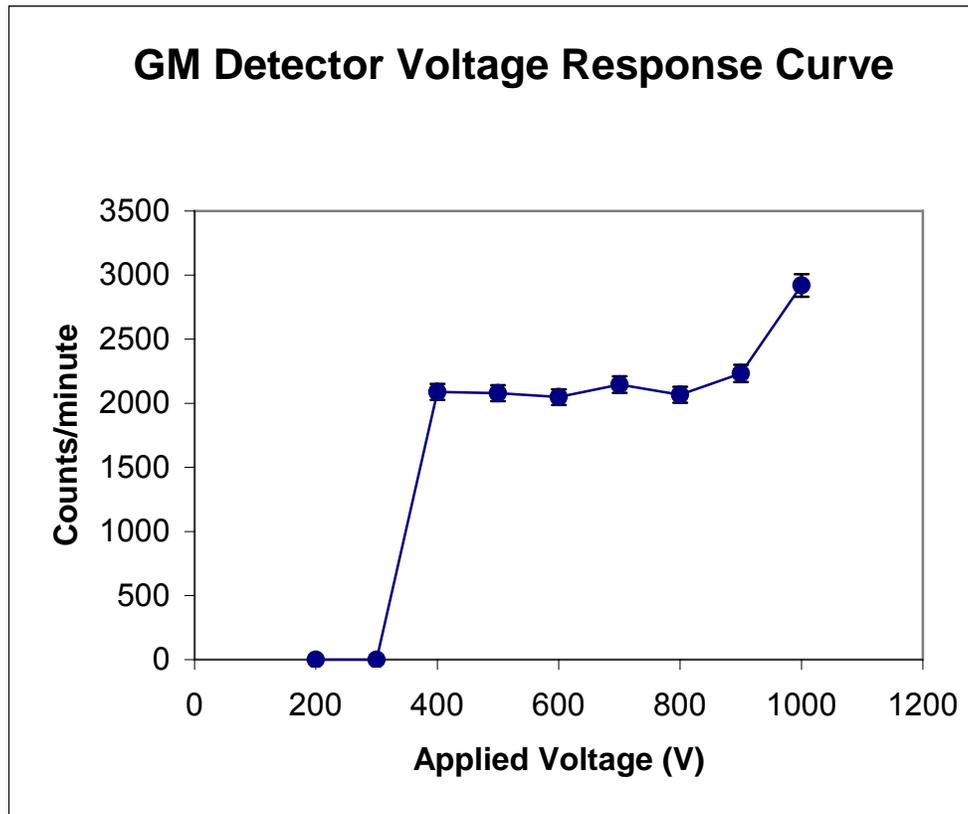
This is the summing up of your argument or experiment, and should relate back to the Introduction and objectives. Outcome(s) of the experiment—was it a success? Discuss the merits and/or pitfalls of the experiment. If appropriate, suggest how to improve the procedure, and what additional experiments or research would be helpful.

References

Cite any references, if any, that you have used outside of the Laboratory Manual.

GRAPHING GUIDELINES

This is a good example of what a graph should look like.



Scale

Proper scaling of graphs is important, not only so that the data fits in the space allocated, but, more importantly, so that the information is clearly conveyed in a visually pleasing manner.

Size: determined by the precision of the information to be conveyed and by the space available.

Type of scale: The two most common scales are linear and logarithmic. As a rule of thumb, when the range of interest of a variable extends over two or more orders of magnitude, then a log scale is used in preference to a linear scale. Log scales are also used to demonstrate an exponential (log-lin) or power (log-log) relationship between two variables. (Conversely, use a linear scale if you want to show a linear relationship.)

Data Points

Size and form: Data points should be large enough and clear enough to be distinguished easily, but not so large as to blot out significant information. When several relationships or several data sources are shown on a single graph, each type of point should have a unique symbol.

Error bars: When the experimental uncertainty of a value is larger than the size of the data point, it **must have an error bar**. (If there are many data points and the uncertainties vary slowly, it is sufficient to give error bars on every nth point.) Error bars in one dimension are usually sufficient, but occasionally error bars in both dimensions are required.

Text and Labels

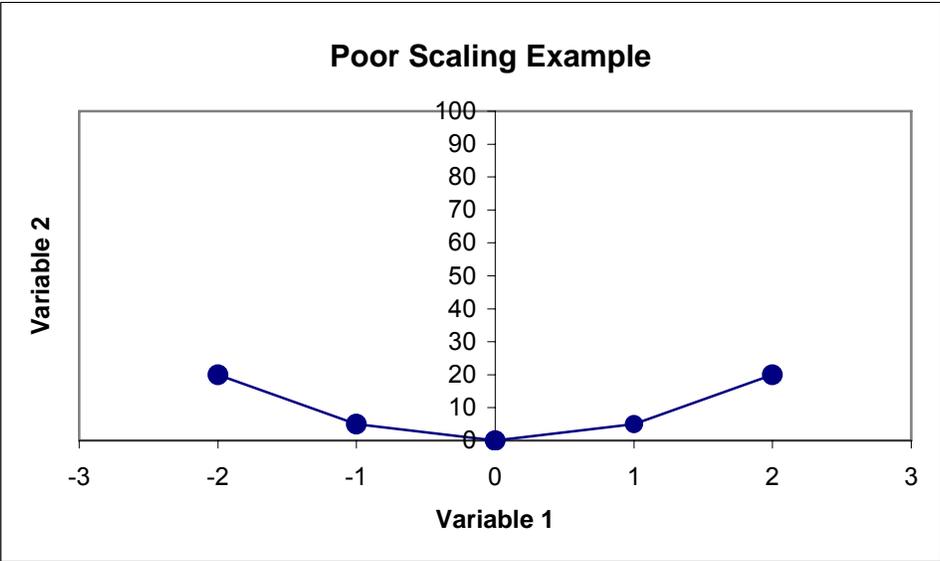
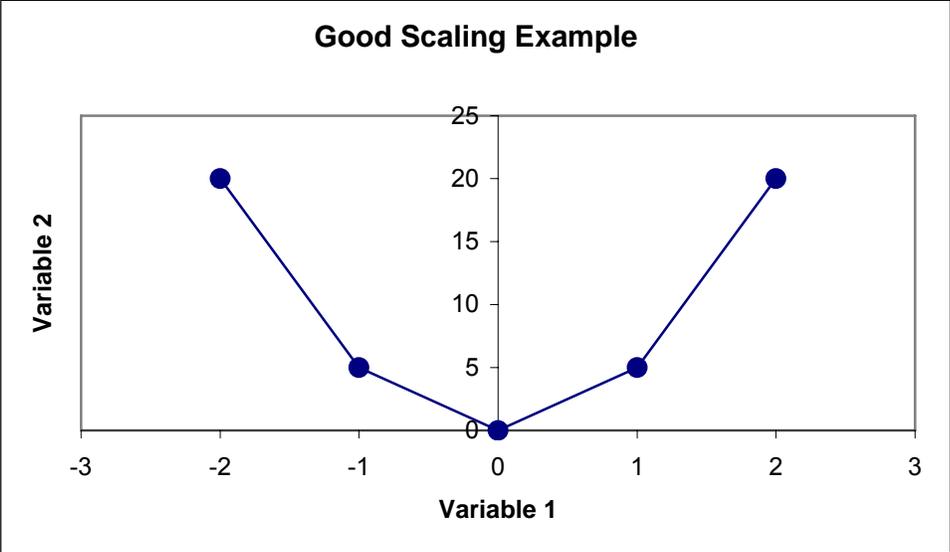
Grid: For most computer-drawn graphs (and graphs prepared for publication) it is preferable to show only tick marks along the axes and omit the grid. (An exception is a graph from which the reader is expected to extract precise values.)

Axes: Each axis must be labeled with its variable name, its dimensions, and the scale.

Data points and curves: All data-point symbols and curves should be clearly identified. A convenient way to do this is with a symbol table or "legend" printed in a vacant region of the graph. An alternative is to identify the data points and curves in the figure caption.

Figure caption: Every figure should have a title, or figure caption that describes briefly the subject of the figure. When there is more than one figure in a report, the captions should include running figure numbers.

Make sure the plots you show in your report clearly illustrate the relationship you intend to show. In the example below, which one clearly shows how Y varies with X:



TABLES GUIDELINES

Reporting Data in Tables

Most often the data to be reported consist of multiple numbers. These should be collected and presented in tables and/or graphs. An example of a partial table for gain measurements is:

Table 1. Gain of the Preamplifier

Pulsar Voltage^a (V)	Pulse Width (Φs)	Output Voltage (V)	Gain (V/pC)
10.00 \pm 0.20	4.00 \pm 0.20	3.29 \pm 0.08	0.86 \pm 0.05
10.00 \pm 0.20	2.00 \pm 0.10	1.53 \pm 0.05	0.80 \pm 0.05
10.00 \pm 0.20	1.00 \pm 0.05	0.87 \pm 0.04	0.91 \pm 0.06
8.00 \pm 0.20	0.400 \pm 0.020	0.250 \pm 0.020	0.82 \pm 0.08
6.00 \pm 0.20	0.400 \pm 0.020	0.200 \pm 0.020	0.88 \pm 0.10

a) Input voltage is pulser voltage \div 10.

Note that:

1. The data (the first three columns) are given together with the analysis (column 4). In a student lab report it is a good idea to state the equations used to calculate the derived quantities (the gain and its uncertainty) and show one calculation with numbers substituted in the equations. (This will earn you partial credit if you make a mistake.) Do not show the calculation for each set of data; give the results in a table as above.
2. **Every** experimental number, and **every** number calculated from an experimental number(s) should have an **uncertainty**. If there is an applicable formalism for calculating the uncertainties, it should be used. This will always be the case for calculated numbers, and for some data values (counting number and averages of multiple measurements, for example.) If all uncertainties in a column are the same, the uncertainty can be included in the column heading rather than repeated for each value. In the above example, the first column could have been headed

Pulser Voltage (V)
(\pm 0.2 V)

and the uncertainties omitted.

3. Round numbers correctly and consistently. For a number with no uncertainty,

rounding should show its approximate precision. When rounding a number with an uncertainty:

- a. Decide how you want to round uncertainties. Common methods are to round the uncertainty to one or two significant figures, or to round to two figures if the error digits are # nn, to one figure if > nn. (The above table was rounded this way, with nn = 25.)
 - b. Round the value to the same number of decimal places as the uncertainty.
 - c. In most cases, a number with zeros for place holders (e.g., 3000±500) should be converted to an exponential [(3.0±0.5)×10³] or cast in different units (see d.)
 - d. Use unit prefixes to give reasonable values. For example, note the use of V/pC (volts per picocoulomb) to avoid giving numbers like 8.64×10¹¹ in column four of the example.
4. The table should have an **appropriate title and column headings**. When there is more than one table in a report, the titles should include running table numbers.

Additional Suggestions:

1. Watch capitalization. Names of elements and chemicals are not capitalized except for the first word in a sentence or a trade name.
2. Refer to supplementary data in your report as Appendix 1, Table 2, and Figure 3. These items are capitalized. Do not spell out the numbers.
3. You may write formulas as H3PO4 or HNO3 rather than H₃PO₄ and HNO₃. However, make sure that you include all relevant charges such as HPO₄(2-) for HPO₄²⁻ or CO₃(2-) for CO₃²⁻.
4. Never start a sentence with a number that is a value.
One sample of HCl was titrated. (**acceptable**)
5.00 mL of HCl was titrated. (**not acceptable**)
5. Make sure that a reference is cited when any data is quoted for comparison with experimental values.
6. Set numerical data apart by using a table if more than several values were obtained. Single values (numbers that are not part of a series and do not belong in a table should be set off on a separate line or boxed. An example [1] is:

The half-life of ¹⁰⁸Ag calculated from the slope of the decay curve in Figure 1 is:

2.4 ± 0.2 min

Watch out for the correct number of significant figures.
Make sure any spreadsheet data is consistently formatted.

Do not bury data in text!

7. The third person impersonal, passive tense is generally used for scientific writing (there is some heated debate in some circles on this point). Remember that you did the experiment in the past, the results may still be true but you are reporting ***what you observed and measured***.

Examples of good and bad writing habits:

The sample was titrated using 0.1000 M NaOH . (***acceptable***)

We titrated the sample using 0.1000 M NaOH. (***not acceptable***)

The sample is titrated by 0.1000 M NaOH. (***not acceptable***)

8. Report the results of your measurements, calculations, and observations. Only report the theory and relevant procedures that would enable a chemist of your ability to reasonably reproduce your work. You don't need to explain how to use a pipette, wipe off a spectrophotometer cuvette, or any incidental procedures unless the experiment could not be repeated without performing that step in detail.
9. References should be cited by number at the first location where the information is used (including paraphrasing). You do not have to cite the reference again unless a direct quote is being used or a key piece of information is required.

Introduction to Gas Ionization Detectors: Geiger-Müller Detectors

OBJECTIVES

1. To understand four important parameters used to characterize the GM Detector, including efficiency, sensitivity, resolving time, and response you will carry out the following tasks:
 - You will set up a GM detector and determine its optimum operating voltage.
 - You will measure the efficiency of your GM detector as a function of the nature of the decay and the energy of the radioactive decay event.
 - You will measure your GM detector's resolving time.
2. To understand the statistical significance of counting data you will carry out the following tasks:
 - You will measure the effects of background radiation and sample counting error on the uncertainty of your "raw" counting measurements.
 - You will demonstrate that counting data will vary statistically approximating a Gaussian distribution.

INTRODUCTION

This introduction is divided into three main parts. The first section will acquaint you with some of the physics of gas-filled radiation detectors, of which Geiger-Müller (GM) detectors are one type. The second section discusses some fundamental properties of all radiation detectors such as detector efficiency and dead time corrections. The third section introduces you to the statistical theory that describes all radioactive counting experiments. This laboratory experiment is extremely important in that the measurement and analysis techniques investigated here are universally applicable to all other types of radiation detectors.

About one hundred years ago it was discovered that radiation rendered gases conductive. The radiation, alpha (α) and beta (β) particles or gamma (γ) rays, interacts with gas molecules causing ionization: positively charged gas ions and their negatively charged electrons. One of the first detectors built (~ 1928) to detect this ionization was the Geiger-Müller (GM) detector. This detector is remarkably sensitive and has a simple construction making it cheap and easy to maintain. The GM detector is still used today, primarily as a radiation monitor. Its use in research was phased out in the late 1940's when the scintillation detector began taking its place. However, it is an excellent tool to use for education purposes since it is so easy (as you will see) to set up and operate.

GAS IONIZATION DETECTOR THEORY

All detectors we will use in the laboratory course operate under the same principle: they detect radiation (α , β , and γ -rays) with $E > 1$ keV by measurement of the energy exchange between the incident particle/photon and the mass of the detector. The GM detector is basically a gas (typically argon at 0.1 atm plus a small amount of quenching gas, discussed below) filled tube, with a thin end window through which the radiation passes (see Figure 1).

The outer shell of the tube acts as a cathode. Along the inner axis is suspended a conducting wire, the anode. As one applies a positive voltage to the anode relative to the cathode, interesting interactions occur between the radiation and the gas medium.

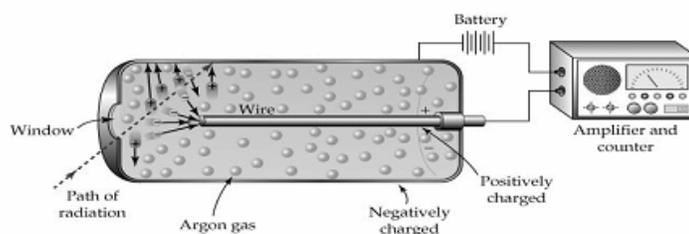


Fig 1: GM Detector

Alpha and beta particles are moving charged particles with electrical fields surrounding them. Their electrical fields interact with gas atoms in the detector by decelerating the particle and accelerating the orbital electrons of the gas molecule. If the energy transfer is sufficient to ionize the gas, an electron-ion pair is created. The average energy lost from the incident particle per ion pair created is called the **W-value** and is slightly larger than the ionization potential of the gas. The W-value is slightly larger than the ionization potential because electronic and vibrational excitation of the gas molecules, in addition to ionization, can also occur during the passage of the particle. The mean number of pairs created is proportional to the energy deposited in the gas. If the gas volume is large (and one of the advantages of a gas detector is that it can be very large) with respect to the mean free path of the incident radiation, the radiation energy is completely deposited in the gas. By applying voltage to the detector, an electric field, generated by the potential difference between the cathode and the anode, will accelerate the electrons to the anode and the gas ions to the cathode. The electrons move much more quickly due to their lower mass compared to the positive ions and it is their collection at the anode that is registered as a pulse by the detector.

The number of electrons collected depends on the intensity of the electric field (the applied voltage) in a rather complicated manner (see Figure 2). With no applied voltage, no electric current would be measured because the ion-electron pairs would quickly recombine before the electrons traveled to the anode. As voltage is applied, increasing the velocity of the electrons/ions, the recombination forces are overcome; the electrons are accelerated and collected by the anode, and current rises with increasing voltage until the point where all electrons generated by each particle are collected before they can recombine. We must also consider the fate of the ionized gas atoms, typically Ar^+ . They eventually are accelerated to the cathode where their collisions with the cathode material would release additional electrons. To prevent this secondary signal, the fill gas has an additional component, usually a halogen molecule like Cl_2 , $\sim 3\text{-}5\%$ of the fill gas. These molecules

absorb energy from the Ar^+ ions, through collisions, thereby reducing the energy of the Ar^+ ions to below the ionization potential of the cathode material. The quenching agent then de-excites by dissociation vs. further emission of electrons. The advantage of using Cl_2 is that after it dissociates, it will eventually recombine to regenerate the quenching agent.

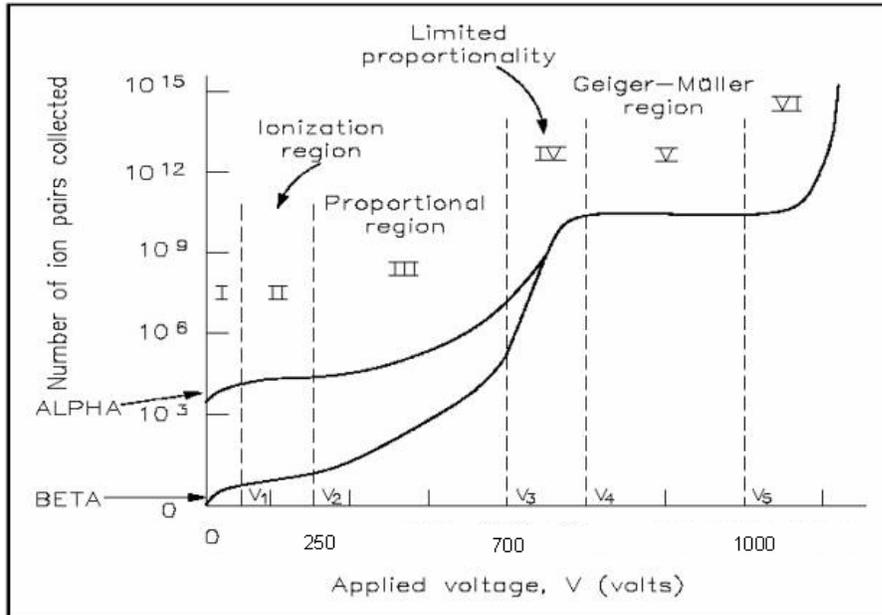


Fig 2: Typical Voltage Response Curve for Gas Ionization Detectors

If one increases the applied potential, the current response is flat with increasing voltage up to ~ 250 V (refer again to Figure 2). A detector working in this region (region II) is called an **ionization chamber**. The signal current is very small and is measured with a device called an electrometer. For example, a 150 keV β particle would be expected to produce, on average, 5000

electrons if the W -value of the medium is 30 eV. The basic energy unit used here is the electron volt (eV). An eV is the kinetic energy acquired by an electron while moving through a potential difference of one volt, and is equal to 1.602×10^{-12} erg or 3.85×10^{-20} calories. Other units used are keV (10^3 eV) and MeV (10^6 eV). Notice the increased current response for α particles compared to β particles in this region. This is because the amount of ionization, I , produced by a charged particle is related to the charge of the particle, Z , its mass, m , and energy, KE :

$$I = \frac{mZ^2}{KE} \quad (1)$$

Since the mass of an α particle is about 7000 times the mass of a β particle, and Z is twice as large, an α particle creates a great deal more ionization per incident particle, hence the increase in current.

If higher voltages are applied to the detector, ~ 250 V - 750 V, one sees again increasing current output (see region III in Figure 2). This additional current is due to production of secondary electrons. The electric field strength, $\xi(r)$, is proportional to the applied voltage, V , and inversely proportional to the distance from the anode, r , by:

$$\xi(r) \propto V/r \quad (2)$$

Therefore, as potential is increased (larger V) large values of the electric field can occur in the immediate vicinity of the anode wire where ' r ' is small. These large electric fields accelerate electrons to energies greater than the ionization energy of the gas molecules. At these energies, electron-gas atom collisions result in further ionization generating new electrons. The electrons produced by this secondary ionization are also accelerated and will interact with the gas to produce still more ionization. This type of ionization is called a *Townsend avalanche* or *cascade*. It occurs very close to the anode wire where the electric field is strongest and in the direction leading to the anode wire (Figure 3). The avalanche terminates when all free electrons have been collected at the anode. The number of electron-ion pairs produced is proportional to the number of primary electrons so in effect, the primary current is amplified by the avalanche (usually by 1000 - 10,000 times). Detectors operating in this voltage range are called **proportional** counters since the gas amplification is proportional to the applied voltage. Since very high field strengths are required to achieve avalanche production, $> 10^6 \text{V/m}$, these detectors are generally of cylindrical design to reduce the voltage requirements.

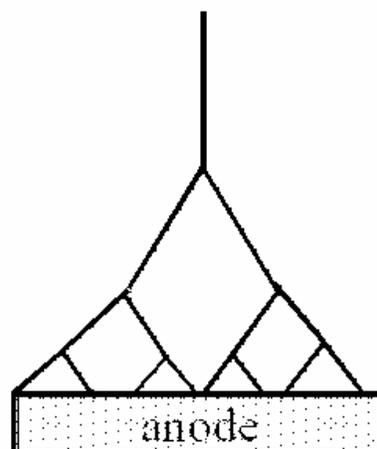


Fig 3: Townsend Cascade

Increasing the voltage further (Region IV, Figure 2) results in additional processes occurring in the detector. Collisions can produce gas ions in excited states ($\sim 11.6 \text{ keV}$) that de-excite by photon emission. These low energy photons travel some distance in the gas before they are absorbed by other gas molecules or the cathode wall (photoelectric absorption). Absorption of these photons in the cathode wall result in the emission of a photoelectron at points far away from the primary event (the ionization potential of metals is much less than Ar). These secondary electrons, if emitted back into the gas region, will also produce avalanches, now all along the tube and the correlation of the resultant signal strength to initial energy deposition becomes non-linear. Detectors are not operated in this voltage region.

CHARACTERISTICS OF GEIGER-MÜLLER DETECTORS

Increasing the voltage beyond 750 V results in a flattened region of current output. In this region(V), the gas medium is completely discharged through a chain of avalanches (Figure 4) instead of just the one avalanche that occurs in the proportional region. This type of discharge always gives the same amplitude, $10^6 - 10^8$ amplification, regardless of the initial energy deposition. This chain of avalanches is called the *Geiger discharge*. In this region of operation it is no longer possible to distinguish between various types of radiation or their energies. The detector is simply a counter of radioactivity. Detectors operated in this energy region, $\sim 750 - 1000 \text{ V}$ are called **Geiger-Müller** Detectors. The presence of the positive gas ions stops the discharge of the detector. Recall that they

move slowly relative to the electrons. Therefore, as the discharge occurs, the number of positive ions produced near the anode is increased, eventually forming a positive charged cloud around the anode. This cloud reduces the electric field and preventing further electron acceleration and generation of new avalanches. At still higher voltages, > 1000V, the tube would continuously discharge (region VI) and the detector becomes damaged. Typically, the GM detectors are operated at a voltage in the middle of the Geiger voltage region.

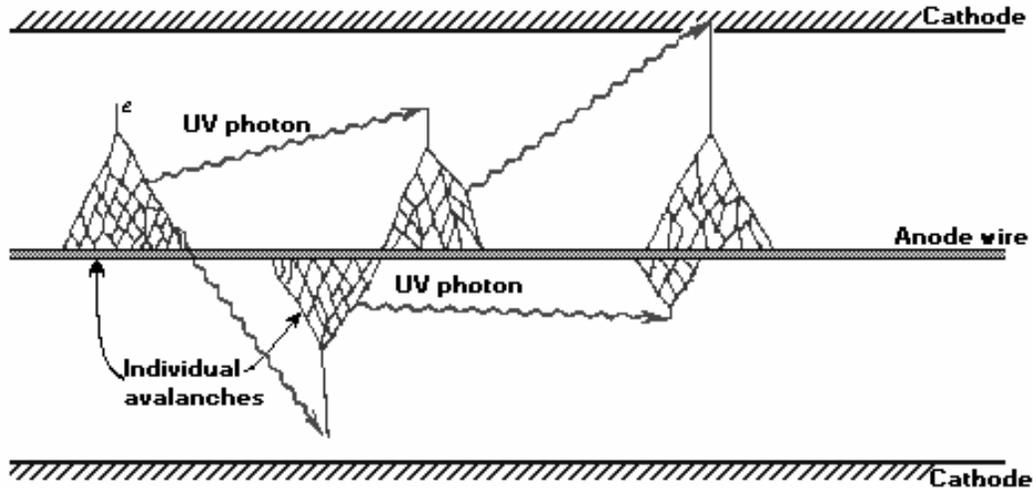


Fig 4: Illustration of a Geiger discharge. Photons originating from excited atoms/molecules within a cascade can photo-ionize.

In summary, the GM Detector is operated at a voltage high enough to completely discharge the detector gas producing the same number of electrons for any incident radiation. The applied high voltage causes a large number of ionizations to occur near the central anode where the electric field is strongest. The positive ions produced form a positive ion cloud around the anode, terminating avalanche production. Hence, each incident particle produces a pulse in the detector. A quench gas is used to prevent a secondary current pulse due to ionization of the cathode by the positive ions.

Finally, there are certain characteristics (see below) of the GM detector that are universal to all radiation detectors. All radiation detectors are based on the transfer of the radiation energy to the detector mass where it is converted to an electrical signal.

1. DETECTOR SENSITIVITY

Sensitivity is the ability of the detector to produce a signal for a given type of radiation with its associated energy. Each detector has its strengths/weaknesses which will be explored in these laboratories. The detector sensitivity depends on:

1. Cross section (or probability) of ionizing reactions in the detector
2. Detector mass
3. Inherent detector noise

4. Material surrounding the sensitive detector volume

The cross section and detector mass determine what fraction of the incident radiation will convert part or all of its energy in the detector into ionization. The ionization signal must be significantly larger than the inherent detector noise to be registered by the detector system.

Finally, the material covering the sensitive volume will absorb some fraction of the incident radiation depending on its type and energy.

2. DETECTOR RESPONSE

The relationship between the incident radiation and the detector output is the detector response. For example, if the detector response (signal pulse height) is linear with energy deposited, it is possible to accurately determine the energy (energies) of the radiation being emitted by the sample. Part of this experiment will be to investigate the GM detector response to different types of radiation. Two other characteristics related to detector response, **energy resolution** and the **response function**, are important when measuring the energy spectrum. They will be considered in later laboratories with detectors other than the GM detectors. GM detectors are not capable of providing any information on the energy of the incident particle.

3. DETECTOR EFFICIENCY

The fraction of the radiation which enters the detector volume and produces a detectable event is called the **intrinsic efficiency** of the detector. Charged particles are highly ionizing. Gamma rays (high energy photons), often emitted in conjunction with α and β particles, produce ionization only with low efficiency since they are not charged. They must first interact with the detector to produce charged particles which are then capable of ionization in the detector medium. In fact, the interaction probability of γ -rays with the detector gas is negligible and any signal due to γ -rays is probably due to their interactions with the walls of the tube to produce photoelectrons which then interact with the gas molecules/atoms. The intrinsic efficiency varies from a few percent for x-rays and γ -rays to near 100% for charged particles for GM detectors.

The ratio of the radiation which the detector registers as decay events to the absolute amount of radiation emitted by the source is the **counting efficiency**. In addition to the intrinsic efficiency, other factors affect the amount of radiation seen by the detector. These include:

1. The solid angle subtended by the detector with respect to the sample.
2. The fraction of radiation self-absorbed by the sample (related to the energy of the radiation).
3. The fraction self-scattered by the sample.
4. The fraction of radiation absorbed by the detector window (related to the energy of the radiation).

5. The fraction of radiation absorbed by air (distance from the source to the detector and related to the energy of the radiation).
6. High Voltage applied to detector.

Hence, the relationship between the observed counting rate for a sample and the corresponding disintegration rate of the radionuclide(s) in the source is not simple. Each factor listed above can contribute to efficiency losses or gains. For example, the efficiency of the end-window GM counter is very low for α particles even though the intrinsic efficiency is 100%. The loss in efficiency results from absorption of the α particles by the air gap between the sample and the detector and absorption of the α radiation by the end window material itself. A detector calibrated with the pure β -emitter, ^{32}P , is not calibrated for another β -emitter, such as ^{14}C , since the β energies are different and the effects of absorption by the sample matrix and the airspace between detector and sample would vary. On the other hand, radiation can be scattered by surrounding material into the detector, increasing the efficiency compared to a geometry that did not contain the scattering material. There are several ways to determine the overall counting efficiency of a detector. The most common method, and the one employed in this experiment, is counting of a source with known disintegration rate, called a **calibrated standard**. The counting geometry includes the shape of the radioactive source, its mass, and its distance from the detector window. For example, if the calibrated standard was 1 μCi (3.7×10^4 dps) and the detector system counted 3.7×10^2 dps, the efficiency factor (ϵ) for the detector would be 0.01 or 1%:

$$\epsilon = \text{Sample Activity/Standard Activity} \quad (3)$$

4. DETECTOR RESOLVING TIME

The resolving time or more commonly, dead time, of a detector is the time during which the detector is responding to one event and can not detect an additional event. The positive ion cloud around the anode must dissipate before a new incident particle interaction can give rise to a new discharge. If an additional event occurs before the electric field about the anode has recovered, it will be lost. The difference between the true and observed count is known as the coincidence loss. Typical dead times for GM detectors are on the order of 200 microseconds. This is a relatively slow device. Ge and Na(I) detectors can have much faster performance with dead times in nanoseconds. Count rates may be corrected for dead time losses using the Equations (10) and (11) given below.

CHARACTERISTICS OF RADIOACTIVE COUNTING DATA

We make several assumptions when analyzing radioactive counting data:

- all the nuclei are identical and independent of each other,

- and each has a definite and constant probability of decay in a unit time interval.

If we make 50 independent measurements of a sample's activity (counts per second, as you will in this experiment), each measurement may differ from all of the others. What is the true value and how do we determine the accuracy and precision of our final determination? If we could measure it directly, we could dispense with statistics! The best we can do though, is estimate it.

Sample Mean: As with any measurement, a large number of individual measurements will fluctuate around an average value in a predictable manner if the only error in measurement is random. The average count, $\langle n \rangle$, for N independent measurements is given by:

$$\langle n \rangle = \frac{\sum_{i=1}^N n_i}{N} \quad (4)$$

where $n_{i=1}$ is the first measurement, etc... and N is the total number of measurements. If we make an infinite number of measurements, $\langle n \rangle$ approaches the true mean value, μ .

Sample Variance: The deviation of any individual measurement from the average is $(n - \langle n \rangle)$ and it should be clear that the sum of deviations should equal 0. The standard deviation is denoted by σ and for a large set of data (infinite),

$$\sigma = \sqrt{\frac{\sum_i (n_i - \langle n \rangle)^2}{N}} \quad (5)$$

Because we typically have a finite set of data to work with we must estimate the standard deviation and instead of σ , we use 's':

$$s = \sqrt{\frac{\sum_i (n_i - \langle n \rangle)^2}{N-1}} \quad (6)$$

Note the difference in the denominator. When N is large, $\sigma \approx s$.

The distributions of measurements about the true mean can be described by a Gaussian distribution. The Gaussian distribution is a very important *class* of statistical distributions. Such distributions are symmetric and have bell-shaped density curves with a single peak about the true mean, μ . To speak specifically of any normal distribution, two quantities have to be specified: (i) the mean, μ , where the peak of the density occurs (also

the “true” value you are trying to measure), and (ii) the standard deviation, σ , which indicates the spread of the bell curve. Figure 5 shows different normal distributions centered about different means. The widths of the distributions are characterized by the standard deviation, σ . An important relationship in radiation counting is that the standard deviation on a data set is equal to the square root of the true mean.

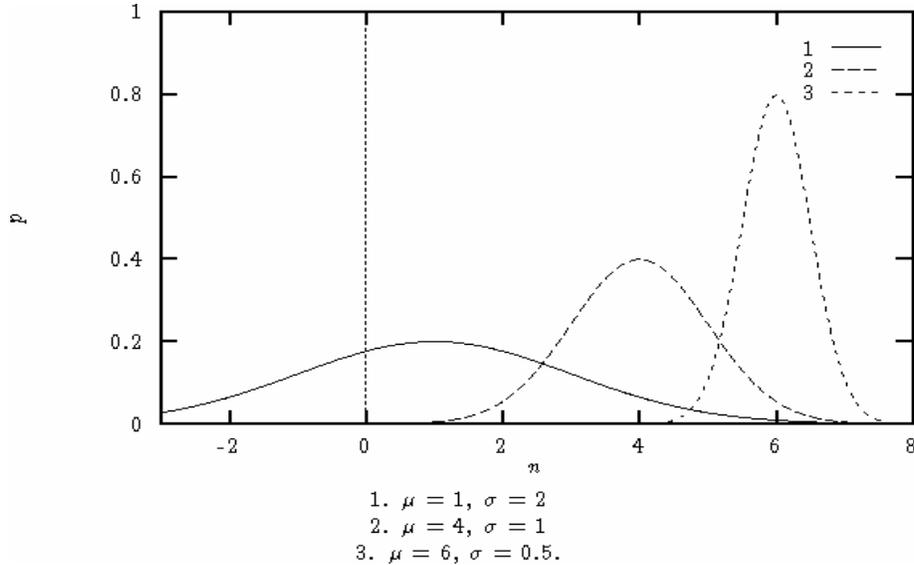


Fig 5: Gaussian distributions as a function of μ and σ . (7)

The height (or probability) of the distribution at any value x is given by:

$$P_G(x, \mu, \sigma) = \frac{1}{\sigma\sqrt{2\pi}} \exp\left[-\frac{1}{2}\left(\frac{x-\mu}{\sigma}\right)^2\right] \quad (8)$$

where the π term serves to normalize the distribution (i.e. make the total probability equal to 1). Although there are many normal curves, they all share an important property that allows us to treat them in a uniform fashion. This property is the **Empirical rule** (see Figure 6) which defines confidence level in the data set at 1 standard deviation, 2 standard deviations and 3 standard deviations:

- 68% of the observations fall within **1 standard deviation** of the **mean**, that is, between $\mu - \sigma$ and $\mu + \sigma$.
- 95% of the observations fall within **2 standard deviations** of the **mean**, that is, between $\mu - 2\sigma$ and $\mu + 2\sigma$.
- 99.7% of the observations fall within **3 standard deviations** of the **mean**, that is, between $\mu - 3\sigma$ and $\mu + 3\sigma$.

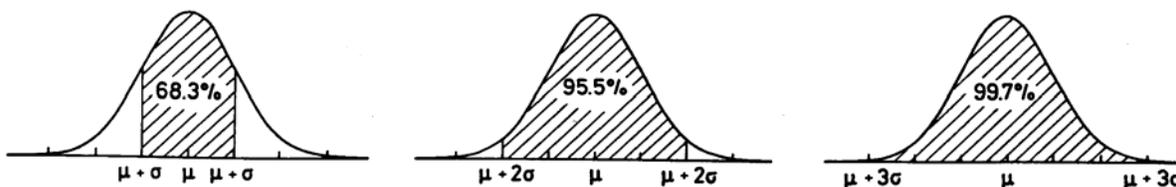


Fig 6: Graphical demonstration of the Empirical Rule for Gaussian distributions.

Thus, for a normal distribution, almost all values lie within **3 standard deviations** of the mean. In practice, one can not exactly measure μ . Thus, the average value, $\langle n \rangle$, is used as the best estimate of μ and the standard deviation is estimated by s . If the observed events are perfectly random, 68.3% of the observed counts should lie between $\langle n \rangle - s$ and $\langle n \rangle + s$. If the sample distribution is truly Gaussian, then the average measurement approaches μ and the value of s approaches σ as the number of observations, N , approaches infinity (but for practical purposes, when N exceeds 100). As an experimentalist, you would desire as narrow a distribution as possible (i.e. a low s value) such that your measurement most closely approximates the true value. When plotting your results, it is customary to include error bars of length \pm sigma on each data point. Examples of determining accuracy and precision of your counting data are given in the Appendix of your Lab Manual.

APPARATUS AND MATERIALS

1. End-window type GM Detector with shelf support for samples (operating instructions are found in Appendix in Lab Manual).
2. A high voltage supply, amplifier, scaler, timer (all in one unit) and backing plates of various materials.
3. Sealed alpha, beta and gamma sources—each team will have a box of appropriate sources to test.
4. Split disk ^{204}Tl source for measuring Resolving Time.

PROCEDURES

In the counting room you will be in an area containing very low levels of radioactive calibrated “sealed” sources that are beta, gamma and alpha emitters. Your Manual Appendices 2-4 contains detailed information on the nature and energy of decay of many of these sources. Also consult your Reference Booklet on Nuclides and Isotopes.

If you are still unclear about operating your GM detector after the pre-lab lecture, consult Appendix 2 (in your Lab Manual) for detailed operating instructions.

Record the detector ID number. For each set of data, record the counting position (shelf number) and standard source identification (radioisotope name, activity, calibration

date).

1. Determining the Characteristic Voltage Curve:

Every GM detector has its own unique voltage response curve. Therefore, the first step when working with a GM detector is to determine its optimum operating voltage by creating a voltage response curve. You should obtain a plateau region and use the approximate middle for your operating voltage. The exact voltage you select is not critical. Selecting the middle of the plateau was a much more important step in the past when stable High Voltage supplies were hard to come by.

- 1.1 Place the counting card, with a β calibrated source of ^{204}Tl at its center on the second shelf of the sample holder beneath the detector. Be careful not to touch the thin window of the detector. **Also note that the source label should be face down.**
- 1.2 Start the counting from the lowest applied voltage. Record data in Table 1.
- 1.3 Increase the voltage in 100-volt steps and count the standard for one minute intervals at each setting. Record the voltages and corresponding count rates (report all count rates in counts per minute or cpm). In the beginning, the count rate is very low; at a certain voltage the rate will begin to rise as the signal pulses from the β particles become large enough to be detected, and the rate will continue to increase until the plateau is reached.
- 1.4 On the plateau, the rate will remain nearly constant (statistically) for successive voltages.
- 1.5 Stop recording data when the counting rate rises 10 to 15 percent above the plateau or you reach 1100 V. **Do not exceed 1100 V.** A good tube should have a plateau slope of less than 10% per 100 volts. Often the slope is as little as 3% per 100 volts. Generate a voltage response curve and calculate the slope on the plateau:

$$\% \text{ per 100 Volts} = \left[\frac{\left(\frac{R_2 - R_1}{R_1} \right)}{V_2 - V_1} \right] \times 100 \quad (9)$$

Label the axes clearly and generate a descriptive title that includes the detector identification number (serial number and vendor). Use this graph to determine your operating voltage—use this value in any further measurements with this instrument.

2. Assessing Data Uncertainty from Background Radiation and Statistical Error:

In many experiments, the activity due to background radiation can be significant. This is especially true when the sample activity is low. Say for example that your background is 30 cpm. If your sample activity is 1000 cpm, the background is only 3% of the sample. However, if your sample activity is 100 cpm, the background is now 30% of the activity! It is good practice to always subtract background from the total count.

- 2.1 Record three consecutive background counts using counting periods of 0.1, 0.5, 1 and 5 minutes and record your data in the attached Table 2. Repeat this process using your gamma source placed on shelf 3. You may tape the data sheet shown at the end of the lab to record the data in your notebook.
- 2.2 Determine the average background count rate and source count rate for each time period. Also determine the standard deviation of each. Finally, calculate the background corrected value at each time period propagating your error.

$$\sigma_{Rb} = \sqrt{\sigma_R^2 + \sigma_b^2}$$

where, σ_{Rb} is the standard deviation of the background corrected count,

σ_R is the standard deviation of the raw corrected count,

and, σ_b is the standard deviation of the background count.

- 2.3 Plot average counts versus time showing the error bars. Be sure to correct all the data to cpm.

3. Measuring Instrument Resolving Time:

As discussed above, the GM Detector is a relatively slow device. At higher count rates, the measured count rate is increasingly less than the true count rate due to dead time losses. When it is used to measure count rates above ~ 5000 cpm, it is probably necessary to make a dead-time correction to obtain the true counting rate. In this section you will record data to determine the resolving time of your detector and subsequently, the dead time correction. Note: It is critical that the counting geometry be the same throughout this part. Any small change in position will change the count rate sufficiently to produce erroneous dead time determinations.

- 3.1 Obtain the split source containing ^{204}Tl from your source box.
- 3.2 Place the right half of the split source on shelf 2 and place the blank source (clear plastic) in the left side. Count for 1 minute. Record your data in Table 3.
- 3.3 Remove the blank (while not disturbing the position of the right side source) and place the left half of the source in with the right half and count for one minute. Record the count.
- 3.4 Remove the right half (while not moving the left half), replace it with the blank source, and count the left half for one minute. Record this count.
- 3.5 Repeat steps 3.1- 3.4 using a lower shelf.

26. Calculate the resolving time (in units of time per count) of the GM tube at the shelf position with the following formula (R_1 = count rate for one half, R_2 = count rate for other half, R_T = count rate for both halves):

$$T_R = \frac{R_1 + R_2 - R_T}{2 R_1 R_2} \quad (10)$$

The true count rate, R , can be determined for an observed count rate, R_0 , using:

$$R = \frac{R_0}{1 - R_0 T_R} \quad (11)$$

Use this equation to correct any count rates that are greater than 5000 cpm and note the correction(s) in your notebook. For example, you might have recorded a data point of 15,000 cpm. You would report this value along with the dead time corrected count of 15,780 cpm (resolving time of detector being 200 μ s).

4. Determining Detector Response to Different Types of Radiation and Energy:

As mentioned in the introduction, the detector counting efficiency is not equal for all types of radiation. From the preceding series of measurements, you should be able to determine which types of particles are best counted with this detector and the effect of particle energy on detector counting efficiency.

- 4.1 Use the **same shelf** (likely shelf 2) for all of your measurements in this section. You will be measuring 1 minute counts for all your sources. Your team source box will be equipped with two gamma sources (either ^{57}Co , ^{109}Cd or ^{60}Co), two beta sources (including ^{14}C and ^{204}Tl), and one alpha source (either ^{252}Cf , ^{244}Cm , ^{241}Am , ^{237}Np or ^{210}Po). You will be performing count measurements on two gamma sources, two beta sources and one alpha source and recording data in Table 4.
- 4.2 Correct all count rates for background and if necessary resolving time (i.e. if the measured count rate is > 5000 cpm).
- 4.3 Convert measured count rate R in cpm to activity (dps):

$$A(\text{dps}) = \frac{R(\text{cpm})}{(60\text{sec/min}) \times \% \text{decay}} \quad (12)$$

Note: This correction is required because detector calibration standards are always provided with activity in units of either μCi or nCi . The curie units can be correlated to disintegrations per second (dps) by the following conversion factor:

$$1 \mu\text{Ci} = 3.7 \times 10^4 \text{ dps}$$

The term “disintegration” applies to all pathways in which nucleus A decays to form nucleus B. However many isotopes decay by pathways not involving emission of the particle being measured. For example, the decay of ^{204}Tl occurs 97.1% by beta decay (which can be measured using a GM detector) and 2.9% by EC (no beta emission). The % decay term in Equation (12) accounts for the competing pathways. The table below provides guidance for decay pathways of the isotopes you will be using in this section of your lab. Further information can be found in your Manual’s Appendix or at the link: <http://isotopes.lbl.gov/education/isotopes.htm>

Isotope	Decay Mode	% Decay Mode	Energies
^{204}Tl	beta	97.1%	763 KeV
^{14}C	beta	100%	157 KeV
^{57}Co	gamma	100%	14.9, 122, 136 KeV
^{60}Co	gamma	100%	1174, 1333 KeV
^{109}Cd	gamma	100%	88 KeV
^{252}Cf	alpha	96.9%	6.2 MeV
^{244}Cm	alpha	100%	5.8 MeV
^{241}Am	alpha	63.1%	5.4 MeV
^{237}Np	alpha	100%	4.8 MeV
^{210}Po	alpha	100%	5.3 MeV

4.4 Calculate the detector’s percentage of efficiency for each isotope you tested by using the following formula:

$$\% \text{ Efficiency} = \frac{A_m(\text{dps})}{A_s(\mu\text{Ci}) \times 3.7 \times 10^4 (\text{dps}/\mu\text{Ci})} \times 100 \quad (13)$$

where $A_m(\text{dps})$ is the your measured activity of the standard converted to dps using Equation (12), and A_s is the calibrated source disintegration rate in μCi (decayed to time of measurement of A_m). To calculate the decay loss of the calibrated source activity from the calibration date to time of

measurement activity, use:

$$A(t) = A_0 e^{-\lambda t} \quad (14)$$

where $A(t)$ is the time corrected activity (A_s in Equation 13), A_0 is the activity at time of calibration (reported on the standard itself), t is the elapsed time, and λ is the decay constant ($\lambda = \ln 2/\text{half-life}$). Be sure the units of elapsed time and half-life are the same.

Example: A student has a calibrated standard of ^{32}P (1.5 μCi on 15-June-2003 at 13:00). She is using this standard to measure the efficiency of a GM detector on 03-July-2003 at 15:30. The decay correction is:

$$A(t=\text{July } 03, 2003) = 1.5 e^{-\left(\frac{0.693}{14 \text{ d}} \times 17.9 \text{ d}\right)}$$

and $A(t) = 0.62 \mu\text{Ci}$ assuming the half-life of ^{32}P is 14 days and the elapsed time is 17.9 days.

4.5 Calculate the efficiencies of your GM detector for all the sources you tested.

POST-LAB DELIVERABLES

Notebook

- Include a brief statement of the lab's objectives.
- Include a sketch of your GM setup for counting samples and label parts.
- Brief description of the procedures.
- Include copies of Tables 1-5. (Note: Tables 3 & 5 should also appear in your final report as it has columns for manipulated data).
- Include a copy of the completed Excel spreadsheet printed to "Fit" on a single page.
- Include any observations from the lab that may have impacted your data.

Final Report

- Include a 1-page summary of what this lab was about (key elements we are looking for include title, date, lab partners, statement of objectives a brief statement on how these objectives were met through a description of the basic principles being demonstrated).

- Include labeled graph of counts vs. voltage
 - show calculations of plateau slope
 - Indicate selected voltage used
- Include labeled graph of counts versus time interval showing error bars.
- Show your calculations of your GM system's resolving time for both shelves used. Show your calculations of the true count rate of a sample using your lowest resolving time if the measured sample activity was 100,000 cpm.
- Include the completed Table 5 and show one sample calculation of your GM counting efficiency using Equations 12-14 to correct for the fractional decay mode of your source (if necessary) and the decay correction of your source with time.
- Include a discussion section of your final results answering the questions below.
- Include a conclusion statement summarizing your general findings. (Were all objectives met?)

Questions for Discussion

1. Discuss why you observe a flattening in your count rate versus voltage graph. What happens with too much voltage is applied?
2. Discuss how instrument resolving time can impact the precision of the counting data you obtain.
3. Discuss what trends you observed in detector efficiency with the nature of the radioactive decay. As a follow-up discuss why is an end-window GM detector not the detector of choice for α and γ radiation?
4. Discuss what trends you observed in your GM efficiency for beta decay as a function of particle's energy. Consider the following beta sources: ^{14}C , ^{33}P , ^{204}Tl , and ^{32}P . What slope would you expect from a curve generated by plotting E_{max} vs efficiency. Postulate a reason for this dependence.
5. Discuss what trends you observed in your GM efficiency for gamma decay as a function of gamma energy. Is the trend the same as with beta decay? Explain your answer.
6. What can you say about the length of time a sample should be counted given your analysis of background counting rates in Table 2 and the graph you generated for this final report.

REFERENCES

1. W.R. Leo, Techniques for Nuclear and Particle Physics Experiments, A How-to Approach, 2nd ed., Springer-Verlag (1994).
2. B.V. Liengme, A Guide to Microsoft Excel, Butterworth Heinemann (2000).
3. F. Kinard, San Jose Summer School Laboratory Experiments
4. P. R. Bevington, D. K. Robinson, P. Bevington, Data Reduction and Error Analysis for the Physical Sciences, McGraw Hill, 3rd Ed. (2002).

Table 1.

Raw Data for Voltage Plateau Measurement

Counts	Voltage
	100
	200
	300
	400
	500
	600
	700
	800
	900
	1000
	1100

Table 2.
Data on Background and Counting Error

Sample	Counting Time (seconds)	Total Counts 1	Total Counts 2	Total Counts 3	Average Counts	Average (cpm)	Standard Deviation
Background	0.1 min						
Source	0.1 min						
Net Counting Rate							
Relative percent Error							
Background	0.5 min						
Source	0.5 min						
Net Counting Rate							
Relative percent Error							
Background	1.0 min						
Source	1.0 min						
Net Counting Rate							
Relative percent Error							
Background	5.0 min						
Source	5.0 min						
Net Counting Rate							
Relative percent Error							

**Table 3.
Resolving Time Data**

Shelf Number	R₁	R₂	R_T	Calculated Resolving Time (μSec)

**Table 4.
GM Efficiency Data**

Background Count Rate _____

			Measured Count Data		Calculated Data		
Radioactive Source	Mode of Decay	Decay Energy	Raw Counts (cpm)	Bkd. Corrected Counts (cpm)	Bkd. Corrects Counts (dps)	Calc. Source Activity (dps)	Detector Efficiency (%)

Elemental Isotope Dilution Analysis

OBJECTIVES

In today's lab you will learn something about the basic principles underscoring Isotope Dilution Analysis (IDA). You will also apply this method to measure the unknown concentration of iron in solution.

BACKGROUND

Isotope dilution analysis (IDA) was one of the first techniques to apply radioisotopes in chemical analysis, having been introduced by Hevesy and Hobbie in 1932 (ref 1). Subsequent to 1940 the usefulness of isotope dilution methods has been reported frequently and is now accepted as an indispensable technique (see references 2 and 3 for reviews) for performing various analyses which would otherwise be extremely tedious or impossible. The technique has become a resource using stable isotopes (for example ^{13}C , ^{15}N and ^{18}O) coupled with mass spectrometry or electromagnetic isotope separators (Isotopic Dilution Mass Spectrometry). Other methods for measuring stable isotope ratios are possible, including optical, neutron activation analysis, and nuclear magnetic resonance. Furthermore, the technique has grown in use with radioactive isotopes and nuclear counting techniques.

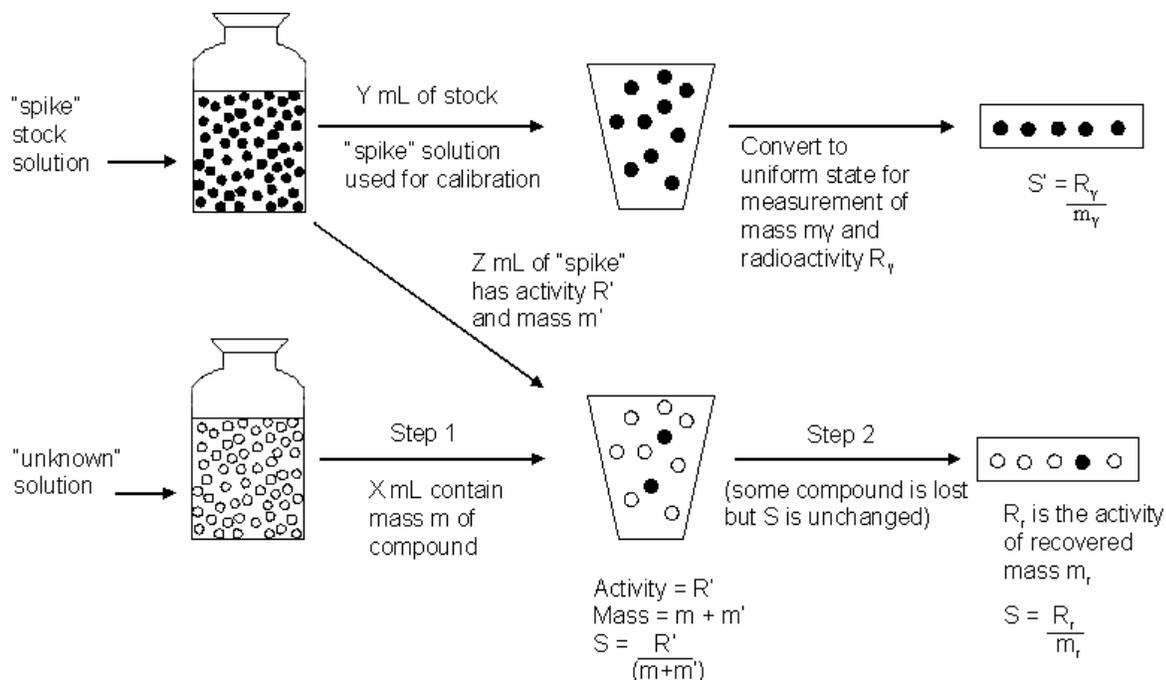
The underlying principle behind the technique of IDA is the **conservation of mass upon dilution**. When radioisotopes are used, the conservation of activity is the manifestation of the conservation of mass principle. The dilution of the radioactive isotope by its non-radioactive counterpart results in the reduction of specific activity, defined as the activity per unit volume or mass, in a conserved manner proportional to the original specific activity and the amount of analyte.

When separated stable isotopes are used, the diluted natural isotopic ratio is related to concentration by the amount and isotopic composition of the separated isotope that was added.

There are three general types of isotope dilution methods. These include: (i) inverse; (ii) direct; and (iii) double isotope dilution. These methods are based on the same fundamental principle, but differ somewhat in their basic approach. What often dictates whether one approach is used over another is the nature of the sample. For example, unknown amounts of radioactive substances can be quantified using inverse IDA where known amounts of inactive (carrier) material are added to the sample. Both the direct and double isotope dilution approaches involve "spiking" an unknown sample with a known amount of isotopically labeled compound (ie. of known specific activity). Double isotope dilution has further merits in mass spectrometric assay. For example, it is possible to quantify amounts of naturally occurring ^{80}Se in a sample using this approach by spiking the sample with a known amount of both ^{78}Se and ^{76}Se enriched stable isotopes and using isotopic ratios between each to arrive at the unknown fraction.

In today's experiment you will determine the Fe content in an unknown using direct IDA. Your spike isotope will be ^{59}Fe and the method used to recover the Fe will be hydroxide precipitation. The techniques involved will consist of the following basic processes:

1. Step 1. Addition of a "spike" consisting of a known amount of isotopically labeled compound with a known specific activity (S') to the unknown mixture containing the same compound made up of stable isotopes. The two are thoroughly mixed to obtain a uniform distribution.
2. Step 2. Suitable treatment of the mixture to isolate the same compound in pure form. **It is essential that the isolated compound be pure, but it is not necessary that all of the compound be recovered in the process.** Thus, it is possible to avoid the tedious processes required for quantitative separations which are otherwise impractical.
3. Step 3. Determination of the isotope content of the isolated portion by measurement of its specific activity (S). The ratio of active and inactive molecules depends on the relative masses of (active) substance added (m') and (inactive) substance originally present (m).



If,

R' = activity (cpm) in spike used for the analytical assay

m' = mass of spike used for analytical assay

m = mass of inactive substance in unknown

then the specific activity of spike S' is given by:

$$S' = R' / m' \quad (1)$$

And the specific activity of the substance isolated in pure form from the mixture is given by:

$$S = R' / (m + m') = R_r / m_r \quad (2)$$

It is desired to know the mass (m) of the substance in the unknown in terms of easily measurable quantities. This may be accomplished by dividing equation (1) by equation (2) and solving for m . Thus,

$$S' = \frac{R' / m'}{R' / (m + m')} = \frac{(m + m')}{m'} = 1 + \frac{m}{m'} \quad (3)$$

$$\frac{m}{m'} = \frac{S'}{S} - 1 \quad (4)$$

$$m = m' [(S'/S) - 1] \quad (5)$$

Consider the Example: A sample of mass (m) was spiked with 0.05 grams of sodium phosphate having an activity of 20,000 cpm.

$$S' = R' / m' = 20,000 / 0.05 = 400,000 \text{ cpm / g}$$

0.1 grams (ie., m_r) of pure sodium phosphate was recovered having an activity of R_r of 600 cpm.

$$S = 600 / 0.1 = 6000 \text{ cpm / g}$$

$$m = m' [(S'/S) - 1]$$

$$m = 0.05 [(400,000 / 6000) - 1] = 3.33 \text{ grams}$$

Merits and Limitations of IDA: The main advantage of IDA is the luxury of using non-quantitative isolation procedures in the separation of analyte. This advantage provides considerable flexibility in the design of separations, which can be optimized for

simplicity, robustness, speed and cost. IDA is not limited to just single element analyses if proper attention is given in tracer design linked to ways to discern the different tracers afterward. The sensitivity of IDA can often be high, but varies according to both the technique used to assess the level of tracer (whether stable isotope or radioactive isotope) and the nature of the tracer isotope itself (ex. looking at oxygen signals by mass spectrometry can be daunting given the background due to water and air in the system). Generally, the limit in IDA is the smallest amount that be determined or purified (this is usually dictated by what can be measured on your analytical instrument for quantifying mass). The principle limitation to IDA is the availability of a suitable spike or tracer. For approaches using radioisotopes, the half-life and type of radiation emitted by the radiotracer is very important, as is its purity. The half-life must be long enough so that sufficient activity is available during final analysis for good counting statistics. However, too long a half-life can be problematic due to low specific activities, storage and disposal of radioactive waste. The type of radiation emitted is important primarily in relation to the ease of measurement. There are suitable ($t_{1/2} > 10$ min) radiotracers available for most elements in the periodic table. The exceptions are He, Li, B, N, O and Ne. Furthermore, separated stable isotopes are available for some 80% of the elements in the periodic table.

APPARATUS

1. 5 mL counting vials (white plastic) and 10 mL centrifuge cones.
2. Heat lamp, planchettes, spatula, tweezers.
3. G-M detector (see Appendix 2 in your Lab Manual)
4. ^{59}Fe stock solution that will contain a **known amount** of FeCl_3 carrier.
5. An “unknown” iron solution (we will provide you will the amount for later analysis).
6. 6 N NH_4OH .
7. Dewars and dry ice.
8. Filter setups with cold traps.

PROCEDURES

Preparation and Calibration of Spike Sample:

1. To a centrifuge cone (test tube) add 1 mL of your spike stock solution. Precipitate $\text{Fe}(\text{OH})_3$ by adding 6 N NH_4OH (**dropwise**) while slowly swirling.
2. Set up filter station. Connect filter flask to cold trap and vacuum. If the cold trap does not have dry ice in it, notify the TA. Anchor the filter flask to a ring stand - make sure it is secure! Test system using H_2O .
3. Pre-weigh the filter paper to the nearest 0.1 mg.

4. Pour some of the precipitate solution to the filter funnel. Start the vacuum. Filter the precipitate. Remember that you do not have to recover all of the precipitate - that is one of the strengths of this method - that it does not depend on 100% sample recovery.
5. **After** all the water has been pulled through the filter, rinse the filter with acetone - use either a pipette or a squirt bottle if available. Dry the paper further under the heat lamp (**use tweezers to handle the filter paper as it is radioactive**).
6. Weigh the $\text{Fe}(\text{OH})_3$ filter sample to the nearest 0.1 mg. **Use weighing paper underneath your sample so as to not contaminate the balance!** Dispose of the weighing paper as radioactive trash.
7. Obtain an Al counting planchette from your bench kit. Affix a small adhesive label off to the side indicating that this is your Standard Spike Sample. Center your filter paper in the planchette and cover the sample with Kapton tape. You will later count this sample using the GM-detector.

Preparation of Unknown Sample using the Spike Solution:

1. To a centrifuge cone (test tube) add 1 mL of an unknown iron solution and 0.2 mL of your ^{59}Fe spike solution. Note that you have added only 1/5 of the carrier Fe (and activity) in your spike solution to the unknown.
2. Precipitate $\text{Fe}(\text{OH})_3$ by adding 6 N NH_4OH (**dropwise**) while slowly swirling.
3. Pre-weigh another filter paper to the nearest 0.1 mg.
4. Use the same filter station you set up for Part 1. Ensure dry ice trap is recharged.
5. Pour some of the precipitate solution to the filter funnel. Start the vacuum. Filter the precipitate. Remember that you do not have to recover all of the precipitate - that is one of the strengths of this method - that it does not depend on 100% sample recovery.
6. **After** all the water has been pulled through the filter, rinse the filter with acetone - use either pipette or bottle if available. Dry the paper further under the heat lamp (**use tweezers to handle the filter paper as it is radioactive**).
7. Weigh the $\text{Fe}(\text{OH})_3$ filter sample to the nearest 0.1 mg. **Use weighing paper underneath your sample so as to not contaminate the balance!** Dispose of the weighing paper as radioactive trash.
8. Obtain another Al counting planchette from your kit and affix a small adhesive label off to the side indicating that this is your Unknown Sample. Center your filter paper in the planchette and cover the sample with Kapton tape. You will later count this sample using the GM-detector.

Workspace Clean-up:

1. Clean work bench area as per instructions from TA. You should be aware of things

that you used in this lab that will need de-contamination before storage (eg., tweezers, filter glassware).

2. You will need to carefully disassemble the filter station—take precaution when removing the cold trap.

Counting Samples:

1. Set up the same GM detector you used in the previous lab. Note what your optimal counting plateau was in that experiment and set the same voltage for today's measurements.
2. Collect a background count with the GM detector - typically 5 minutes should be adequate.
3. **Count each sample with and without a thin aluminum absorber** (~ 200 mg/cm²) between the sample and the counter. The absorber permits only the gammas to be counted. Record the detector used and the shelf used (use same shelf through out). Collect > 2000 counts each time you count. Note your count interval and use it to convert your raw counts to counts per minute (cpm). Do three consecutive measurements of your count rate and record your cpm's and counting error in Table 1 located at the back of this lab.

DATA ANALYSIS

Assuming the radioactive spike solution to contain a known amount of iron as FeCl₃ per volume of solution, you will calculate the mass of Fe⁺³ (m) in your unknown sample in mg/mL using Eq. 5). Also remember you need to correct for the fact that iron present in the initial solution is FeCl₃ (you will be told the molar concentration of this spike solution) and what you weigh out is present as a precipitate is Fe(OH)₃. You will also, calculate the precipitation yields for both your spike stock solution and the unknown solution (based on your measurement of mass (m) above). For the spike solution this will be the following:

$$\% \text{ Precipitation Yield Spike} = \frac{(\text{mass of Fe}^{+3} \text{ in sample weighed})}{(\text{calculated mass of Fe}^{+3} \text{ you introduced})} \times 100$$

For your unknown solution this will be the following:

$$\% \text{ Precipitation Yield Unknown} = \frac{(\text{mass of Fe}^{+3} \text{ in sample weighed})}{(\text{your calculated mass of Fe}^{+3} \text{ or "m" })} \times 100$$

POST-LAB DELIVERABLES

Notebook

- Include a brief statement of the lab's objectives.
- Include a sketch of your experimental setup.
- Include a brief (1 paragraph) procedure.
- Include copies of Tables 1 a & b for your raw data.
- Show a record of mass measurements.
- Include any observations from the lab that may have impacted your results.

Lab Report

- Include a 1-page summary of what this lab was about (key elements we are looking for include title, date, lab partners, statement of objectives a brief statement on how these objectives were met through a description of the basic principles being demonstrated).
- Include completed copies of Tables 1 & 2.
 - show all your calculations for error propagation, specific activities, and final mass calculation of your "unknown" from both sets of data (ie. with and without absorber).

Useful Data Analysis Tips: the TA's will provide you with the molar concentration of FeCl_3 in your spike solution. You will need to calculate how many mg of Fe^{+3} there are per mL of your spike solution (use MW $\text{FeCl}_3 = 162.2$ g/mole with Fe^{+3} having a fractional abundance of 0.344). Furthermore, you will be weighing $\text{Fe}(\text{OH})_3$ precipitate not FeCl_3 . (MW $\text{Fe}(\text{OH})_3 = 106.9$ g/mole with Fe^{+3} having a fractional abundance of 0.523).

- Show calculations of your precipitation yields for both your spike solution and "unknown" solution based on data collected with and without the aluminum absorber.
- Include a discussion section of your final results answering the questions below.
- Include a conclusion statement summarizing your general findings. Were all objectives met? How accurate was your measurement of the "unknown" iron sample? Consider your results with and without the aluminum absorber. Was self attenuation of radioactivity a problem? If not why?

Discussion Questions

1. Based on your precipitation yields, discuss the merits of the IDA approach for elemental analysis (ie. explain why the amount sampled does not matter in the use of IDA.)
2. List two assumptions that should be evaluated during the design of a radiotracer experiment (you may consult your text on this). For this particular IDA experiment, discuss how each assumption was fulfilled.
3. Earlier you were told that IDA using radiotracers is typically limited to single element analyses, but not necessarily exclusive. Consider now a sample that contains unknown amounts of both Fe(III) and Sn(II) cations. Also consider that both Fe(OH)₃ and Sn(OH)₂ possess solubility constants at 20°C of 2.79×10^{-39} and 5.4×10^{-27} , respectively. **Hence, both hydroxides will precipitate together.** You have at your disposal three possible radiotracers to use, but you are told that you can limit yourself to using only **two** of these, and **both** must occupy one spike stock solution.

Isotope	Half-life	Decay Properties
⁵⁹ Fe	44.5 day	0.46 MeV β ⁻ 1.1 & 1.21 MeV γ's
¹²¹ Sn	27 hr	0.38 MeV β ⁻ only
¹¹⁰ Sn	4 hr	0.283 MeV γ only

You are given the amount of carrier associated with each radiotracer you use (**ie. both m'_{Fe} and m'_{Sn} are known**).

Using the above information, describe one approach that would allow you to effectively carry out an IDA for individual mass measurements of Fe(III) and Sn(II) in your unknown sample. Note, there is more than one way to approach this problem. Hint: first write out variations of equation 5 for Fe and Sn contributions (ie. treating each as separate problems). The challenge of course is to devise a method that will allow you to dissect the total activities S' and S down into measurable terms of individual isotopic contributions (ie. S'_{Fe} , S_{Fe} and S'_{Sn} , S_{Sn}). Also consider the characteristics of each isotope's mode of radioactive decay and half-life in your approach.

REFERENCES

1. G. Hevesy and R. Hobbie (1932) Z. Anal. Chem. 28:1-32.
2. Radiochemistry and Nuclear Methods of Analysis, Ehmann and Vance, Wiley and Sons, Inc. (1991) pp. 318-323.
3. J. Tolgyessy, T. Braun and M. Kyrs (1972) Isotope Dilution Analysis, Pergamon Press, Oxford.

Table 1a. Raw Count Data with Absorber

Sample Label	Count 1 (cpm)	σ	Count 2 (cpm)	σ	Count 3 (cpm)	σ
Spike						
"Unknown"						

Table 1b. Raw Count Data without Absorber

Sample Label	Count 1 (cpm)	σ	Count 2 (cpm)	σ	Count 3 (cpm)	σ
Spike						
"Unknown"						

Table 2a. Specific Activity Calculations with Absorber

Sample Label	Mass Fe(OH) ₃ in mg	Average Count (cpm)	Average Count Error	Specific Activity S = R/m	Specific Activity Error
Spike Stock	(m _y)	(R _y)		(S')	
Unknown	(m _r)	(R _r)		(S)	
Spike Mass				(m')	

Table 2b. Specific Activity Calculations without Absorber

Sample Label	Mass Fe(OH) ₃ in mg	Average Count (cpm)	Average Count Error	Specific Activity S = R/m	Specific Activity Error
Spike Stock	(m _y)	(R _y)		(S')	
Unknown	(m _r)	(R _r)		(S)	
Spike Mass				(m')	

Interaction of Radiation with Matter

OBJECTIVES

From Rutherford's earliest experiments exploring the radiation of uranium, it became obvious that not all radiation penetrated matter in the same way. The interactions of the various radiations with matter are unique and determine their penetrability through matter and, consequently, the type and amount of shielding needed for radiation protection.

Being electrically neutral, the interaction of gamma rays with matter is a statistical process and depends on the nature of the absorber as well as the energy of the gamma. There is always a finite probability for a gamma ray to penetrate a given thickness of absorbing material and so, unlike the charged particulate radiations (beta or alpha) which have a maximum range in the absorber where all are stopped regardless of source strength, some gammas will always get through and, given a strong enough source, a lot may get through.

Charged particle radiations (beta and alpha) behave very differently and can be stopped by an appropriate amount of shielding. In fact, because of the large mass of an alpha particle, its radiation can be easily stopped by the thinnest of absorbers. For instance, even an inch of air is sufficient to stop essentially all alpha particles. As a result, sources of α radiation OUTSIDE your body cause very little harm because air and the dead outer layers of your skin protect your living cells inside. However inhaling, eating, or drinking a source of α radiation can kill tissue, cause mutations, and cause cancer.

In today's lab you will carry out the following objectives:

1. You will measure the respective transmission rates of two different energy β particles through various aluminum absorbers. You will construct calibration curves from each using the absorption characteristics of known radioactive sources and known thicknesses of the aluminum absorbers. Furthermore, you will construct a range-energy curve using transmission data compiled from your classmates.
2. Using the calibration curve of beta transmission from one of your sources you will measure the thickness of an unknown aluminum foil.
3. You will measure the transmission rate of a single γ source using lead absorbers.
4. You will measure the transmission rate of a single α source in air using vacuum bell jars that will enable you to remove the air from the path of the alpha particles. You will construct a range-energy plot using stopping pressure data compiled from your classmates.

INTRODUCTION

In the past, data from radiation transmission rates was used to establish some of the basic properties (ie. γ energies and β endpoint energies) of newly discovered radionuclides, and to establish general relationships between particle energy and the absorbing power of different materials. Much of this information was important to better design appropriate shielding for the protection of workers in the field. Today, radionuclides are also widely used in many industrial applications to measure thickness. If you do a web search of **thickness gauging**, you will see the diverse applications and the various sources used. A straightforward application is the measurement of the thickness of rolled steel. Radionuclide methods are quite successful even though the steel temperatures could be 1000 °F and the sheet is moving at up to 40 mph out of the rollers. Another not so typical application is the measurement of fuel left in a tank in rocket travel. In the absence of gravity one certainly can not use a level indicator. The radioactive thickness gauge will tell the amount of fuel even when it is not dispersed uniformly in the tank based on knowledge of absorption of radiation and its dependence on the electron density of the material.

BETA PARTICLES

Range-Energy Relationship

The attenuation of beta rays by any given absorber may be measured by interposing successively thicker absorbers between a beta-ray source and a suitable beta-ray detector, such as a Geiger counter, and counting the beta particles that penetrate the absorbers. When this is done with a pure beta emitter, it is found that the beta-particle counting rate decreases rapidly at first, and then, as the absorber thickness increases, slowly. Eventually, a thickness of absorber is reached that stops all the beta particles; the Geiger counter only registers background counts due to environmental radiation.. Plotting the log of the counts as a function of the absorber thickness will yield a curve much like that shown in Figure 1.

The endpoint of the absorption curve, where no further decrease in the counting rate is observed, is called the **range** of the beta rays in the material of which the absorbers are made. As a rule of thumb, a useful relationship is that the absorber half-thickness (that thickness of absorber which stops half of the beta particles) is about one-eighth the range of the beta rays. Since the maximum beta-ray energies for the various isotopes are

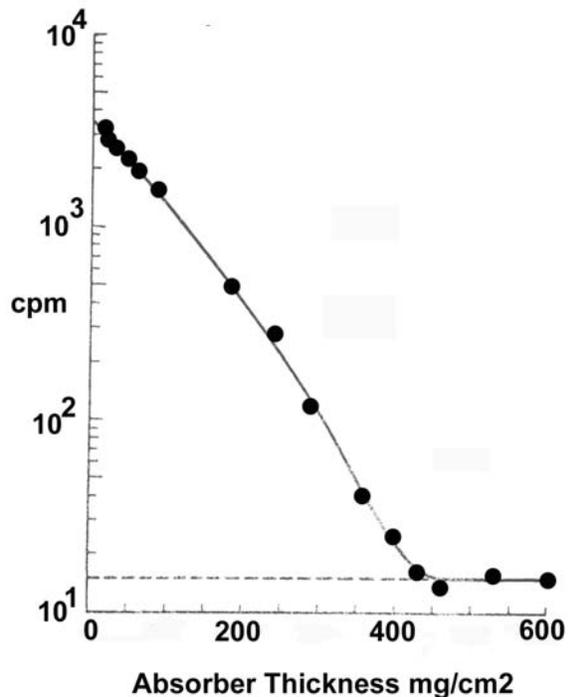


Fig. 1 Absorption curve (aluminum absorbers) of ^{210}Bi beta particles, 1.17 MeV. The dotted line reflects the mean background counting rate.

known, then by measuring the beta-ray ranges in different absorbers, the systematic relationship between range and required thickness of absorber for any given beta energy decreases as the density of the absorber increases (Figure 2).

Detailed analyses of experimental data show that the ability to absorb energy from the beta rays depends mainly on the number of absorbing electrons in the path of the beta ray—that is, on the **areal** density (electrons per cm^2) of electrons in the absorber; and, to a very much lesser degree, on the atomic number of the absorber. For example, the absorbing power of aluminum of 6 mg/cm^2 would be approximately the same as that of air at 6 mg/cm^2 . For practical purposes, therefore, in the calculation of shielding thickness against beta rays, the effect of atomic number is neglected. Areal density of electrons is approximately proportional to the product of the density of the absorber material and the linear thickness of the absorber, thus giving rise to the unit of thickness called the **density thickness**. Mathematically, density thickness t_d is defined as:

$$t_d (\text{g/cm}^2) = \rho (\text{g/cm}^3) \times t_1 (\text{cm}) \quad (1)$$

The units of density and thickness in Eq. 1, of course, need not be grams and centimeters; they may be any consistent set of units. Use of the density thickness unit, such as g/cm^2 or mg/cm^2 for absorber materials, makes it possible to specify such absorbers independently of the absorber material. For example, the density of aluminum is 2.7 g/cm^3 . From Eq 1 a sheet of aluminum 1 cm thick, therefore, has a density thickness of:

$$t_d = 2.7 \text{ g/cm}^3 \times 1 \text{ cm} = 2.7 \text{ g/cm}^2$$

If a sheet of Plexiglas whose density is 1.18 g/cm^3 is to have a beta-ray absorbing quality very nearly equal to that of the 1-cm-thick sheet of aluminum, its linear thickness can be found from Eq. 1:

$$t_1 = t_d / \rho = (2.7 \text{ g/cm}^2) / (1.18 \text{ g/cm}^3) = 2.39 \text{ cm}$$

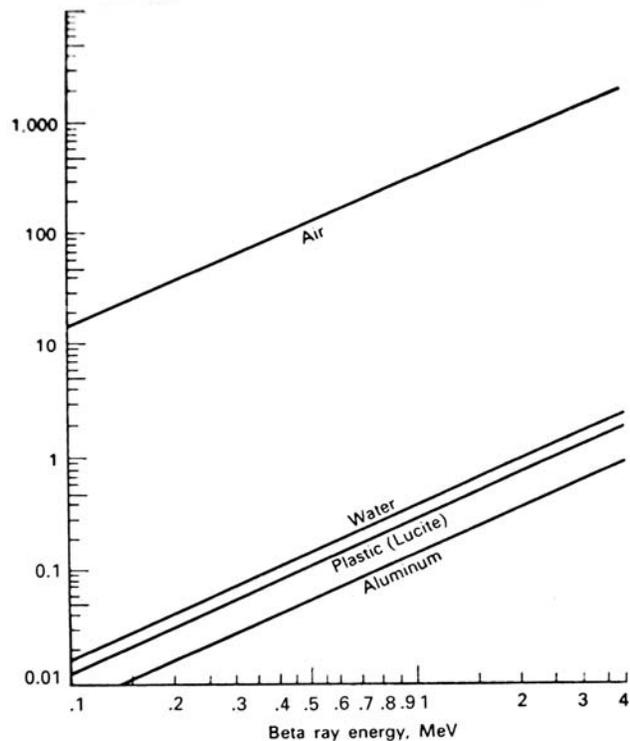


Fig. 2 Range-energy curves for beta rays in various substances.

Another practical advantage of using this system of thickness measurement is that it allows the addition of thickness of different materials in a radiological meaningful way. A universal curve of beta ray range (in units of density thickness, mg/cm^2) versus beta energy is shown in Figure 3.

Today you will generate such a curve from the range-energy data you acquire and compile from all your classmates.

This curve above is fitted by the following general equations:

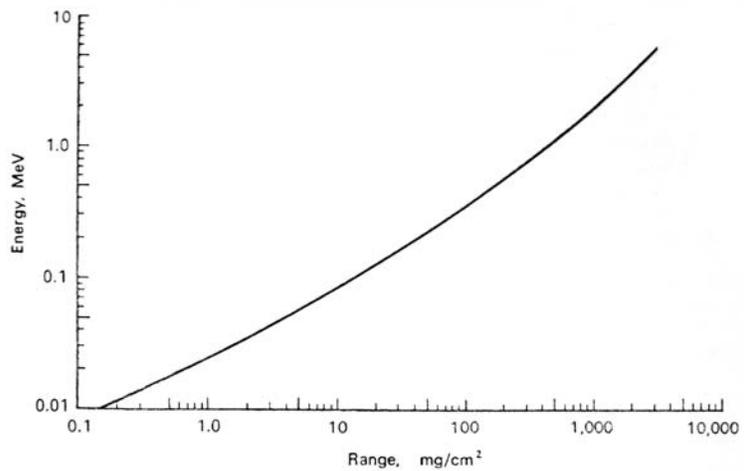


Fig. 3 Range-energy curve for beta particles. The range is expressed in units of density thickness.

$$R_{\max} (\text{mg}/\text{cm}^2) = 412 \times E^{(1.265-0.0954 \ln E)} \quad \text{for } 0.01 \leq E \leq 2.5 \text{ MeV} \quad (2)$$

$$R_{\max} (\text{mg}/\text{cm}^2) = 530E - 106 \quad \text{for } E > 2.5 \text{ MeV} \quad (3)$$

Example:

What must be the minimum thickness of a shield made of (a) Plexiglas and (b) aluminum in order that no beta rays from a ^{90}Sr source pass through?

Solution:

Strontium-90 emits a 0.54 Mev beta particle. However, its daughter, ^{90}Y , is radioactive and emits a beta particle whose maximum energy is 2.27 MeV. Therefore, the shield must be thick enough to stop the more energetic particles. From Figure 2 the range of a 2.27 MeV beta particle is found to be 1.1 g/cm^2 . The density of Plexiglas is 1.18 g/cm^3 . From Eq. 1 the required thickness is found to be:

$$t_1 = t_d / \rho = 1.1 \text{ g}/\text{cm}^2 \div 1.18 \text{ g}/\text{cm}^3 = 0.932 \text{ cm}$$

Using the same equation we find that the required thickness of aluminum to stop all the beta rays is 0.41 cm.

Mechanisms of Beta Energy Loss

1. Ionization and Excitation

Interaction between the electric fields of a beta particle and the orbital electrons of the absorbing medium leads to electronic excitation and ionization. Such interactions involve inelastic collisions. The electron is held in the absorber material atom by electrical forces, and energy is lost by the beta particle in overcoming these forces. Since electrical forces act over long distances, the “collision” between a beta particle and an electron occurs without the particles coming into actual contact—as in the case of the collision between like poles of two magnets. The amount of energy lost by the beta particle depends on its distance of approach to the electron and on its kinetic energy. However, by virtue of their small mass, their penetration into matter is considerably greater than other, heavier, charged particles (ie. alpha particles). Furthermore, because their masses are identical to that of the scattering electrons of the absorber material, their path may take large deviations (Figure 4) and, therefore, even thin absorbers will attenuate beta particles to a large extent.

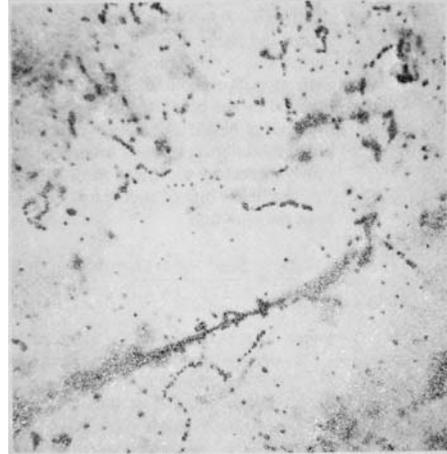


Fig. 4 Electron tracks in photographic emulsion. The tortuous lines are the electron tracks: the heavy line near the bottom was made by an oxygen nucleus in primary cosmic radiation.

2. Radiative Energy Loss

Bremsstrahlung consists of X-rays emitted when high-speed charged particles suffer rapid acceleration. When a beta particle passes close to a nucleus, the strong attractive coulomb force causes the beta particle to deviate sharply from its original path. The change in direction is due to radial acceleration, and the beta particle, in accordance with classic theory, loses energy by electromagnetic radiation at a rate proportional to the square of the acceleration. This means that the bremsstrahlung photons have a continuous energy distribution that ranges downward from a theoretical maximum equal to the kinetic energy of the beta particle.

An almost infamous mode of energy loss is Cerenkov radiation. It is the source of the blue-white glow of very radioactive material immersed in water. It occurs when very fast charged particles enter an absorber of different refractive index (eg. water). The particle energy will exceed the speed of light in that medium and the result is Cerenkov radiation (named after the Russian scientist who investigated it extensively in the 1940's). When the beta particles pass rapidly through water, their electric and magnetic fields excite the water molecules. These molecules then emit part of their energy in the form of light—Cerenkov radiation.

ALPHA PARTICLES

Range-Energy Relationship

Alpha rays are the least penetrating of the radiations. In air, even the

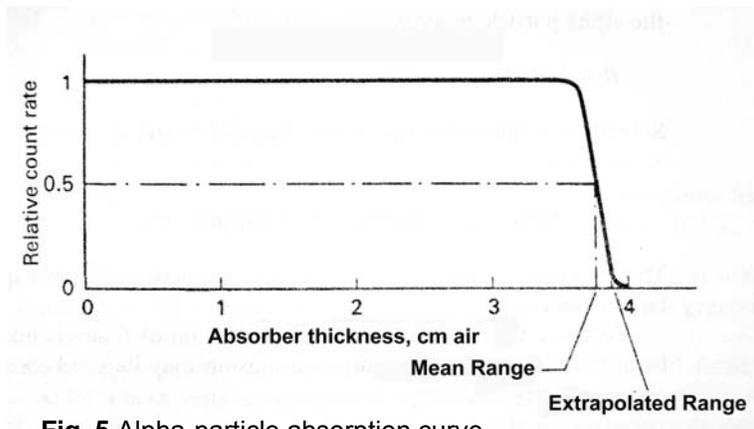


Fig. 5 Alpha-particle absorption curve.

most energetic alphas from radioactive substances travel only several centimeters, while in tissue, the range of alpha radiation is measured in microns (1 micron = 10^{-4} cm). The term **range**, in the case of alpha particles, typically refers to the mean range as depicted in Figure 5.

An alpha particle absorption curve is flat because the alpha radiation is essentially monoenergetic (unlike beta radiation). Increasing thickness of absorbers serves merely to reduce the energy of the alphas that pass through the absorbers; the number of alphas is not reduced until the approximate range is reached. At this point, there is a sharp decrease in the number of alpha particles that pass through the absorber. Near the end of the curve, the absorption rate decreases due to straggling, or the combined effects of the statistical distribution of the average energy loss per ion and the scattering of the absorber nuclei. The **mean range** is the range most accurately determined and corresponds to the range of the average alpha particle. The **extrapolated range** is obtained by extrapolating the absorption curve to zero alpha particles transmitted.

Air is the most commonly used absorbing medium for specifying range-energy relationships of alpha particles. The range of alpha particles in air at 0° C and atmospheric pressure can be approximated from the following equations:

$$R \text{ (cm)} = 0.56 \times E \text{ (MeV)} \quad \text{for } E < 4 \text{ MeV} \quad (4)$$

$$R \text{ (cm)} = 1.24 \times E \text{ (MeV)} - 2.62 \quad \text{for } 4 < E < 8 \text{ MeV} \quad (5)$$

The range of alpha particles in any other medium may be computed from the following relationship:

$$R_m \text{ (mg/cm}^2\text{)} = 0.56 A^{1/3} \times R \quad (6)$$

where,

A = atomic number of the medium

R = range of the alpha particle in air (cm units)

Example:

What thickness of aluminum foil, density 2.7 g/cm^3 , is required to stop the alpha particles from ^{210}Po ?

Solution:

The energy of the ^{210}Po alpha particle is 5.3 MeV. From Eq. 5, the range of the alpha particle in air is

$$R_{\text{air}} = 1.24 \times 5.3 - 2.62 = 3.95 \text{ cm}$$

Substituting this value for R into Eq. 6 and 27 for the atomic number, A, for aluminum we have

$$R_m = 0.56 \times 27^{1/3} \times 3.95 = 6.64 \text{ mg/cm}^2$$

The thickness of this foil, in centimeters, is calculated from Eq. 1 and is found to be 0,00246 cm. Notice how thin the aluminum foil is that stops the alpha particle relative to the thickness needed in the earlier example.

Today, you will construct a range-energy plot by compiling data from your other classmates. Teams of three students each will work with one alpha source and compiling data from four sources will enable you to plot the stopping air pressure for four different energies.

Mechanisms of Alpha Energy Loss

The major energy-loss mechanism for alpha particles is interaction with electrons in the absorbing medium. These interactions result in electronic excitation and ionization of the absorber atoms. In passing through air an alpha particle loses, on average, 35 eV per ion pair that it creates. Because of its high electrical charge and relatively low velocity due to its great mass, the specific ionization of an alpha particle is very high, on the order of tens of thousands of ion pairs per centimeter traveled in air.

GAMMA RAYS**Exponential Absorption**

Unlike charged particles, gamma-rays have no charge and no mass. They are a form of electromagnetic radiation, also called photons. They interact weakly with absorbing material and hence their ranges are much longer than charged particles like alphas or betas. The following is a brief summary of the interactions of photons with matter:

1. Photoelectric Effect: $\propto \frac{Z^5}{E_\gamma^{3/2}}$
2. Compton Effect: $\propto \rho / E_\gamma$ (where ρ is the absorber density)
3. Pair Production: $\propto \log(E_\gamma)$

Since the absorption effect is exponential in nature, there is no defined range of photons in materials. We may study the absorption of photons by materials by measuring the intensity ratio of photons transmitted through the material much as we did with beta particles. The big difference here is that the photons are monoenergetic and their interaction with the absorber atoms is probabilistic and that a single interaction removes the photon from the beam. Obviously this probability depends on the number of nuclei in the material, i.e. its thickness, Δx , so:

$$\text{Probability a gamma interacts with a nucleus (electron)} = \mu_l \Delta x \quad (7)$$

where μ_l is the probability per unit length of an interaction (also called the **linear attenuation coefficient**). Recall that there are various mechanisms for photons to transfer energy to mass and each process would have its own linear attenuation coefficient. To calculate the total μ_l , one would simply add the contributing coefficients of the different processes (i.e., photoelectric effect, Compton effect and pair production). Using Equation (7), one can derive an equation for the transmission probability of a beam of photons through a rectangular slab of absorber (or linear attenuation):

$$I = I_0 e^{-\mu_l t} \quad (8)$$

Because the gamma transmission curves are derived from probability theory and are fundamentally exponential, one can substitute the **mass attenuation coefficient**, μ_m , for the linear attenuation coefficient:

$$\mu_m = \mu_l / \rho \quad (9)$$

and then Equation (8) becomes:

$$I = I_0 e^{-\frac{\mu_l}{\rho} \rho t} = I_0 e^{-\mu_m d} \quad (10)$$

The mass attenuation coefficient is constant regardless of the density of a material (think of ice vs. steam) while the linear attenuation coefficient changes with density. Note also that the product of ρt is now the mass thickness, d , usually represented as mg/cm^2 . **Unlike charged particles, a certain percentage of gammas will always make it through the absorber** (see Figure 6) and it is useful to consider the half-value thickness of a given absorbing material for the gamma ray energies of interest. The half-value thicknesses are

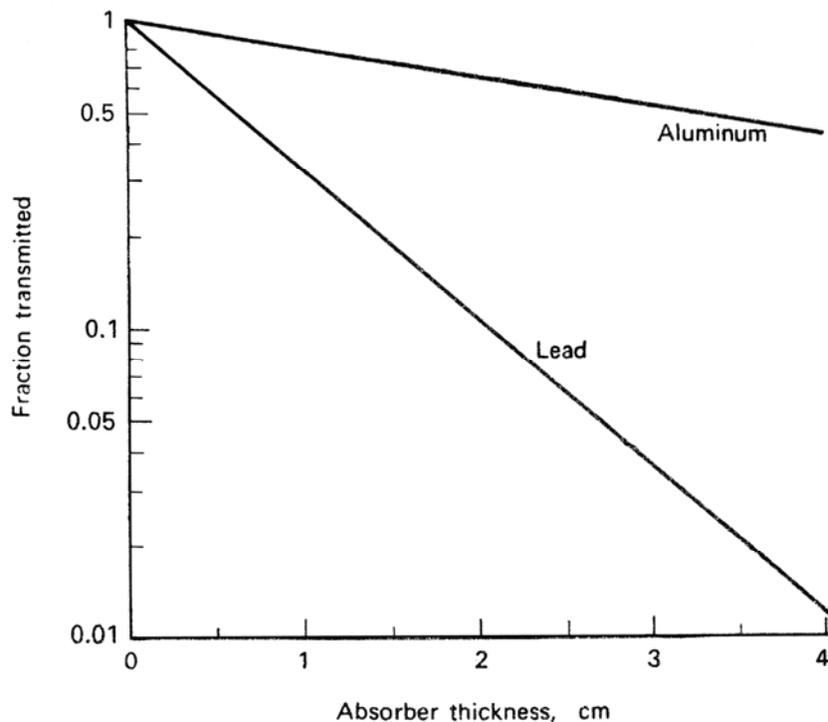


Fig. 6 Attenuation of gamma rays in aluminum and lead absorbers.

determined from Equation 10 using linear attenuation or mass attenuation coefficients found in the literature.

Example:

If the activity of the source is $5.0 \times 10^6 \text{ } \gamma/\text{s}$ with $E_\gamma = 600 \text{ keV}$ incident on a lead sheet of 2 cm thickness, how many gammas will pass through the lead?

Solution:

First, one must look up the mass attenuation coefficient for lead for a gamma with energy of 600 keV. This value happens to be $0.125 \text{ cm}^2/\text{g}$. The density of lead is 11.35 g/cm^3 . Then from Equation (9), $\mu = 0.125 \times 11.35 = 1.42/\text{cm}$ (make sure units are equivalent so they cancel in the exponent term!). Using Equation (8) we have:

$$I = 5 \times 10^6 \text{ } \gamma/\text{s} \exp^{(-1.42 \times 2)} = 2.92 \times 10^5 \text{ } \gamma/\text{s} \text{ pass through (or } \sim 5.2\%)$$

APPARATUS AND MATERIALS

1. End-window Geiger-Müller detector.
2. Beta sources: ^{204}Tl , ^{14}C , ^{36}Cl , ^{137}Cs (**each team will do only two sources**).
3. Gamma source: ^{60}Co .
4. Set of Al and Pb absorbers of known thicknesses.
5. One unknown Al absorber (note the absorber number in your report).
6. Vacuum bell jar, hand vacuum pump and electronic manometer.
7. Alpha sources: ^{241}Am , ^{252}Cf , ^{244}Cm , ^{237}Np (**there will be only one source to test per team**).

PROCEDURES

Part 1. Beta Transmission Curves

1. Set (or determine and set) the voltage of the Geiger-Müller detector to its operative voltage (refer to Operating Instructions found in the Appendix of your Lab Manual). You should be able to use the same voltage you determined in Lab 1.
2. Take three 5-minute background counts.
3. Place one of your beta sources on shelf No. 2 and count the source for 1 minute or 2000 counts. Place the first aluminum absorber on shelf No. 1, measure the count rate again. Repeat the measurements with different absorber thicknesses to characterize a transmission curve. **Be sure to record 3 points beyond the point where all betas are stopped by the absorber. (Refer to Fig 1)** Count long enough to achieve less than 10% uncertainty or 5

minute counts (whichever is shorter). Also note if you are using ^{14}C as a beta source you will need to test thinner Al absorbers than what is provided in your set—the TA's will give you these absorbers.

4. Construct a beta transmission curve plotting Log (cpm) on Y-axis (background corrected) versus absorber thickness (mg/cm^2) on X-axis. Fit the curve using Excel “trend” and display the equation and R^2 fit on your graph.
5. Repeat procedures 3-4 with your other beta source.
6. Construct a range-energy plot by compiling the range data from your other teammates (**you should have range data from four different beta sources with some overlapping data**). Plot Log E_{max} on Y-axis versus Log Range (mg/cm^2) on X-axis.

Part 2. Measuring the Thickness of an Unknown Aluminum Absorber

Obtain an unknown Al absorber from the TA and place it on shelf No. 1. (Note the absorber number in your notebook and final report.) Place one of your beta sources on shelf No. 2 and measure the count rate such that there is $\sim < 10\%$ uncertainty. (Remember the uncertainty is the square root of the count rate so 1000 cpm will have an uncertainty of ± 32 cpm or 3.2%.) Use your beta transmission curve and the equation from the best fit trend line in Excel to determine the unknown absorber thickness.

Part 3. Gamma Transmission Curves

1. Place your gamma source on shelf No. 2. Count long enough to obtain 1000 counts.
2. Place the first Pb absorber on shelf No. 1. Measure the count rate again. Count long enough to achieve less than 5% uncertainty (but do not exceed 5 minute counts).
3. Repeat step 2 with different Pb absorbers to characterize the transmission curve for that source.
4. Construct a plot of gamma transmission by plotting Log (cpm) on the Y-axis (background subtracted, including error) versus absorber thickness (mg/cm^2) on the X-axis.
5. Fit the data and calculate the slope of the line. Calculate the mass attenuation coefficient and the linear attenuation coefficient from your data. Compare these values with the literature extrapolated from Figure 7.
6. Using your experimental linear attenuation coefficient, calculate the half-value thickness (in g/cm^2) for your gamma source in lead.

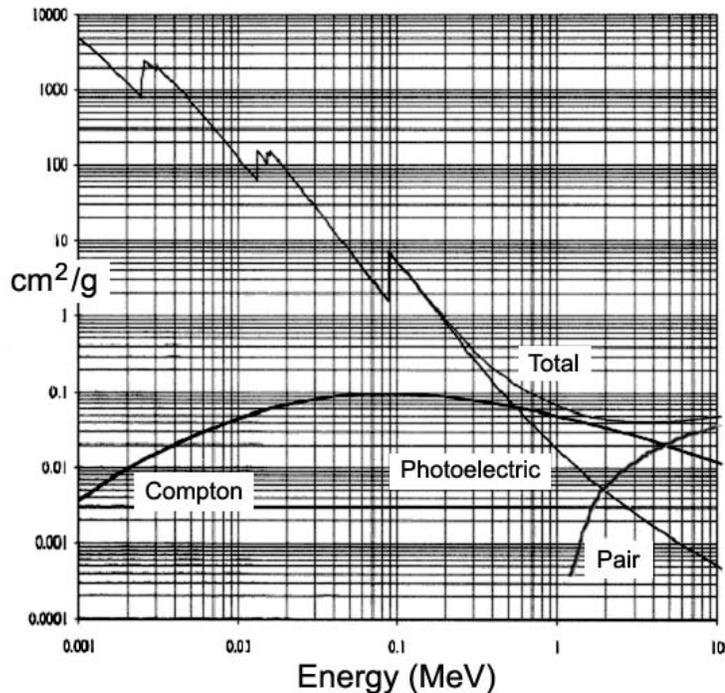


Fig. 7 Mass attenuation coefficients for gamma rays in lead. Multiply by $\rho = 11.35 \text{ g/cm}^3$ to convert values to linear coefficients.

Part 4. Measuring the Range-Energy Relationship of Alpha Particles in Air

1. Each team will have one alpha source. Note the alpha energy of that source in your report.
2. Connect the BNC of the Geiger-Müller detector sealed into your vacuum bell jar to your scaler. Note the operating voltage posted on the side of the bell jar. Set that voltage on your scaler and record the value used in your notebook. Also note the sensitivity correction factor in your notebook.
3. Pump air from the chamber stopping at each 50 torr interval below atmosphere to record a **3 minute count rate** (the counts will be low!). Roughly note the gauge pressure on the electronic manometer where you “see” a change in the count rate. Continue pumping air from the chamber until you hit roughly 400 torr (this will register as 360 torr on your gauge: $760 - 360 = 400$; we will assume atmospheric pressure to be 760 torr). Once you have reached your minimum pressure begin venting air back into the chamber using the trigger handle on your PVC hand vacuum pump. Slowly vent air until you achieve a pressure just slightly under the range you observed a change in the count rate. Collect a few more count rates at 25 torr intervals in this critical range of pressure. Your goal is to target the **mean range pressure** (in torr) like that depicted in Fig. 5.
4. When you have completed 3 slowly vent the chamber to ambient pressure, open the jar and remove the alpha source. Reposition

the jar and collect a background count for the same 3 minute interval.

5. Construct a plot of the background corrected cpm versus chamber pressure (in torr). Included error bars on your data. Record the mean pressure where you observed a change in count rate. For the purposes of this lab you can ascertain the mean range visually from your graph.
6. Compile mean range pressures from the other teams and construct a range-energy graph by plotting $\text{Log}(E_\alpha)$ versus mean stopping pressure (in torr).

POST LAB DELIVERABLES

Notebook

- Include a brief statement of the lab's objectives.
- Include a sketch of your experimental setup.
- Include a brief procedure.
- Include copies of any raw data Tables.
- Include any observations from the lab that may have impacted your data.

Lab Report

- Include a 1-page summary of what this lab was about (key elements we are looking for include title, date, lab partners, statement of objectives a brief statement on how these objectives were met through a description of the basic principles being demonstrated).
- Your lab report should have final graphs and calculations including:
 1. Two (2) beta transmission curves
 2. One beta range-energy curve
 3. Calculation of unknown absorber thickness
 4. Gamma transmission curve
 5. Calculation of mass attenuation and linear attenuation coefficients for your gamma source
 6. Calculation of half-thickness value of Pb needed for your gamma source
 7. Alpha transmission curve
 8. Alpha range-energy curve

- Include a discussion section of your final results answering the questions below.
- Include a conclusion statement summarizing your general findings. Were all objectives met?

Questions to be addressed in your report summary

1. Enumerate possible sources of “background” counting rates. Consider different types of radiation and their penetrating ability from your experiences in this lab. Also, consider if any background might be attributable to the source.
2. Consider how best to plan your measurement times in order to obtain the most precise values of the background corrected rates. Recall Lab 1 which addressed statistical aspects of counting.
3. Discuss any trends you observed in the beta range-energy relationship you observed between the different beta sources. How does your graph compare with Figure 3? What effect does beta energy have on its range in aluminum? What might you do differently to get a better match to theory?
4. Consider that ^{137}Cs undergoes beta decay to an excited state of ^{137}Ba , which in turn decays to the ground state of ^{137}Ba with emission of a 0.662 MeV gamma ray. Is it possible to determine the range of betas emitted from ^{137}Cs by the method used in this experiment?
5. Discuss similarities or differences in your alpha transmission curve relative to that shown in Fig 5. Do you see a step in the count rate at some critical pressure? Explain why you would expect to see a step in count rate using alpha particles, but not using beta particles (think about the shape of one of your beta transmission curves with Al). Do you observe residual counts from your alpha source even at higher pressures? Explain what might cause this effect—think about the decay modes of some of these sources to explain your results—are they pure alpha sources?
6. Discuss any trends you observed in the alpha range-energy relationship you observed between the different alpha sources tested. How does your graph compare with that of the beta range-energy plot you generated earlier? Are the trends the same?

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(NCSS Lab 4)

Nuclear Pulse Instrumentation, Pulse Height Analysis (PHA) and Gamma Spectroscopy using NaI Detectors

OBJECTIVES

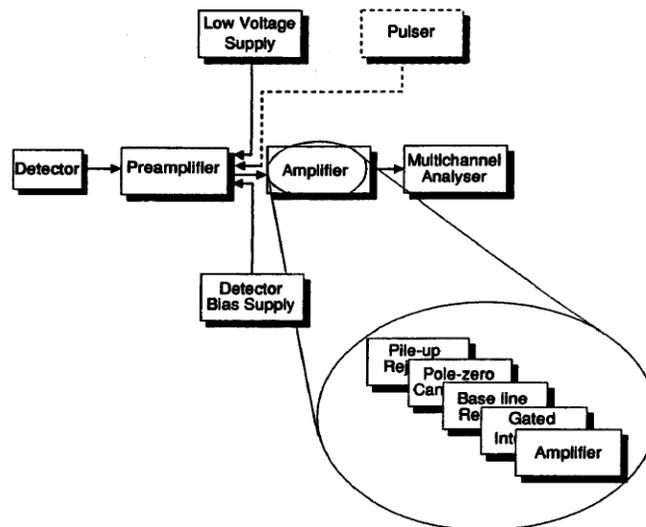
The objective of today's lab is to become familiar with nuclear counting instrumentation and to learn about how the basic components of a pulse-height analyzer can impact signal. A second objective is to see how PHA can be put into practice in gamma spectroscopy using a NaI scintillation detector. Here you will learn how pulse instrumentation translates into a useable spectrum of information with certain energy resolution and detector efficiency.

INTRODUCTION

Detector systems can provide more information on the detected radiation than a simple "count". Gas Ionization Detectors (Geiger-Müller detectors) are basically counters, providing ~1 V pulses for each incident particle. The detectors used in today's lab provide information on the energy that the incident particle deposits in the detector as well. The price one pays for this additional information is that these systems require sophisticated electronics: electronic instrument modules that are connected and their performance optimized to handle the detector generated electrical pulse signals and that will provide the desired output (e.g. energy and number of pulses).

Figure 1 shows a typical schematic for a gamma (γ) spectroscopy counting system.

Fig. 1: A simple schematic electronic system for gamma spectroscopy [2], p. 63.



We could have provided you with a system that was already put together, having performed all necessary “troubleshooting” ourselves, and requiring no thought on your part. The objective for this laboratory though is for you to familiarize yourself with a variety of electronics, like oscilloscopes, but primarily with nuclear pulse instrumentation used in γ -spectroscopy and its capabilities/limitations so that you can put together your own system and optimize its performance to give you the best possible data. It is very important that you fully understand this laboratory since several future laboratories in this course will require you to put together your own nuclear pulse system for measuring environmental samples.

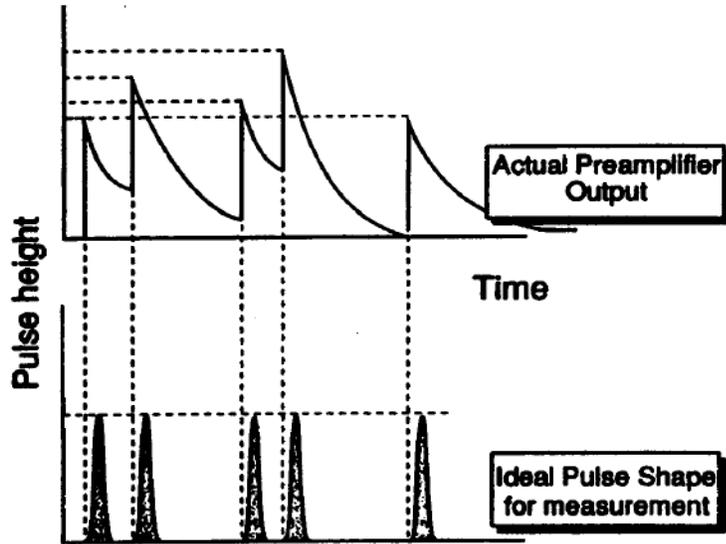
You will be using your detector systems to study γ -ray emissions from various isotopes. You will be studying γ -ray spectroscopy in more detail in a future lab using an intrinsic solid-state germanium detector. The basic principle of operation relative to PHA is the same. For today you only need to know that most isotopes that decay by γ -ray emission do so uniquely such that their spectrum is unique to a particular radionuclide, like a “fingerprint”. The detector you will be using today is a solid scintillator detector (as compared to the gas detector used in GM lab). The detector material is a large crystal (2" or 3" cube) of NaI encased in an Al can. This material has a much higher efficiency for γ -rays than the gaseous GM detectors have (typically $\sim 7\%$ at 662 keV for a 3" cubic crystal and only slightly less for smaller crystals). Gamma-rays interact with the scintillator producing pulses of light that are converted to current pulses in the attached photomultiplier tube.

PULSE GENERATION AND SHAPING

Most detectors operate by collecting the charge (electrons) produced by the radioactive particle’s interaction with the detector. This charge is collected as a current pulse and passed to the **preamplifier (preamp)**, which converts the current pulses to voltage pulses whose amplitudes are proportional to the amount of current collected in the detector. The preamp also has an output stage that is suitable for driving signals through long coaxial cables (which pass the pulses from the preamp to the amplifier, e.g. the black BNC cables). These pulses must then be amplified, in a controlled way, to an easily measured value, and shaped to eliminate extraneous electronic noise. This is the job of the **amplifier (amp)**.

Figure 2 shows a typical preamp output (on top) and the subsequent shaped pulses coming out of the amp (at the bottom). However, the pictures are not to scale. The preamp pulses are typically in the mV range while the amp pulses are 0.25 - 10 V. The amp shapes pulses into a Gaussian form while still retaining the proportionality between the amplitude and the amount of energy deposited by the incident γ -ray on the scintillator. You will monitor the shapes of the amp output pulses by observing their voltage trace on an **oscilloscope**. *Review Appendix 7 for oscilloscope operation.*

Fig. 2: Preamplifier output and the desired converted pulses [2], p. 73.



PULSE HEIGHT ANALYSIS

As the name implies, the pulse heights of the amplifier output pulses can be measured and correlated to the energy deposited by the γ -rays in the detector. This is accomplished in the present experiment using a **multichannel analyzer (MCA)**. This device contains an **analogue-to-digital converter (ADC)** which digitizes the analogue pulse sent from the amp and then passes the digitized pulse to a memory buffer, the multichannel buffer, **MCB**. The memory buffer catalogues the pulses into memory units called bins or **channels** – basically a glorified bean counter. The number of channels is specified by the user. For example, if the memory, which spans 0-10 V (10 V is typically the highest amplification the amplifiers we use are capable of delivering to the MCB)

is divided into 100 channels, a pulse height of 2.5 V would correspond to channel 25.

Once the channel number has been determined, the MCA then adds a count in that channel. After the pulses have all been processed, the experimenter has a graph of counts vs. channel number which is the γ -ray spectrum which might look something like Figure 3. Data acquisition and simple data manipulations are performed by a computer and are based on a commercial software program, ScintiVision®, which is interfaced with the PC serving as the MCA. The instructors

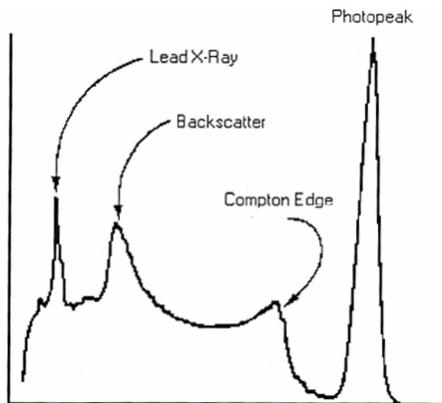


Fig 3: Pulse height spectrum for a single photon emitter using a NaI detector.

will help you become familiar with this program during this experiment. *Review Appendix 8 for MCAs.*

APPARATUS

1. NaI(Tl) detector coupled to a photomultiplier tube (PMT), placed in a shielded enclosure..
2. NIMBIN, coax cables (BNC cables).
3. Base for the PMT with a voltage divider, connected to a stable ripple-free high voltage supply.
4. Preamp with connectors for signal input, signal output, and test signal input (this will be contained within the detector housing).
5. Spectroscopy-grade amp with multi-channel analyzer system (MCA), and an oscilloscope.
6. An arrangement for reproducibly positioning radioactive samples in front of the detector, at various distances (shelves).
7. A set of radioactive γ -ray standards.
8. A computer folder for saved spectra—or you can save on your memory stick.

A block diagram of the scintillation detector system you will put together is shown in Figure 1. The components are described further here. More detailed discussions can be found in the references.

1) NIMBIN and cables: NIMBIN components comprise an industry standard for interchangeable modules. NIM stands for Nuclear Instrumentation Modules which fit into a BIN. This standardization allows one to use modules from different manufacturers to be matched or mixed as required. The BIN contains a rack for mounting the components and also provides them with electric power (± 6 , ± 12 , and ± 24 V). The cables used to connect the bin components to each other and the detector are coaxial cables with BNC connectors. The connectors slip on to the fittings and then are turned about 1/4 turn clockwise to secure them. To take them off, turn the connector 1/4 turn counter-clockwise and carefully remove them.

2) High Voltage Supply: These detector systems require a ~ 1000 V potential across the photomultiplier tube (PMT). The HV unit supplies stable high voltage (typically 0-5000 V) to the PMT. It may have several output terminals. Choose the terminal that can deliver 1000 V (generally there are two terminals depending on which voltage range you desire). Ignore the INH terminal, if there is one. The HV terminals look like the terminals on other modules (amps, ADCs) but they only accept coaxial high voltage cable. On the front panel is an on/off switch.

Before you turn on the NIMBIN, make sure the HV switch is in the OFF position. This is very important since applying voltage to the detector when the detector preamp is not powered will destroy the preamp. Internally there is a switch to change the polarity to positive or negative which is shown by a light on the front. The instructor will change the polarity if it is required. The NaI detectors require positive voltage but verify by examining the detector housing or manual where the operating voltage and polarity are specified. There is also a dial to adjust the high voltage. The instructor will show you how to read this dial since some are not straight forward. **Before you apply HV for the first time, the instructor must inspect your cable connections and settings.**

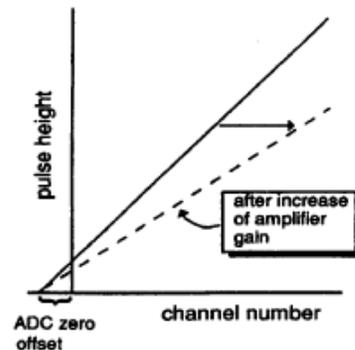
3) Preamplifier: The preamp is within the detector housing (Al can) to reduce signal to noise ratio (which is dependent on the cable length). It has an output and input connector. Preamps get their power from a 9 pin connector (looks like RS-232) that is at the back of the detector housing. They are powered by connecting the power cable to the power outlet on back of the amp. **Note: Connect the preamp power cable to the amp before you turn on the HV supply.**

4) Amplifier: The amp has several outputs depending on what type of signal is to be generated. Use the unipolar output for this lab. Unipolar means the output rises to a maximum value and then returns to zero volts. On the front of the amp is a polarity switch which tells the amp what polarity the preamp pulses are. Select positive polarity. There are two gain dials on the front to adjust the amplification of the preamp signal. There is a pole/zero adjust screw and a BLR adjust screw. These are used to help shape the pulse. The gain is usually adjusted to amplify the 1332 keV pulse from ^{60}Co to $\sim 8\text{ V}$ (as observed on an oscilloscope) or the 662 keV pulse from ^{137}Cs to $\sim 6\text{ V}$. There is also a shaping time switch. This is the time the amp takes to collect and shape the voltage pulses from the preamp. **The longer the shaping time, the more accurate the amplification of the pulse.** Shorter shaping

times are used with more radioactive sources to minimize the dead time (time when the counting system is busy analyzing one pulse and cannot accept another pulse). The effect of amplifier gain is shown in Figure 4. An increase in gain decreases the energy width per channel while a decrease in gain

Figure 4

The ideal response of an MCA, showing ADC zero off-set and the effect of increasing the gain of the amplifier [2], p. 86.



increases the energy width per channel.

5) Detector: A scintillator detector containing a 2" NaI crystal will be used in this laboratory to detect γ -rays. Figure 5 shows a basic schematic of such a detector setup. To measure the energy and number of photons, this detector must be able to detect and differentiate the photons. In other regions of the electromagnetic spectrum, one may use a diffraction grating which uses the wave nature of light to disperse the photons according to their wavelength. The wavelength measured can then be related to the photon energy using $E = hc/\lambda$. Today's lab makes use of a technique called **scintillation** to measure the photon's energy.

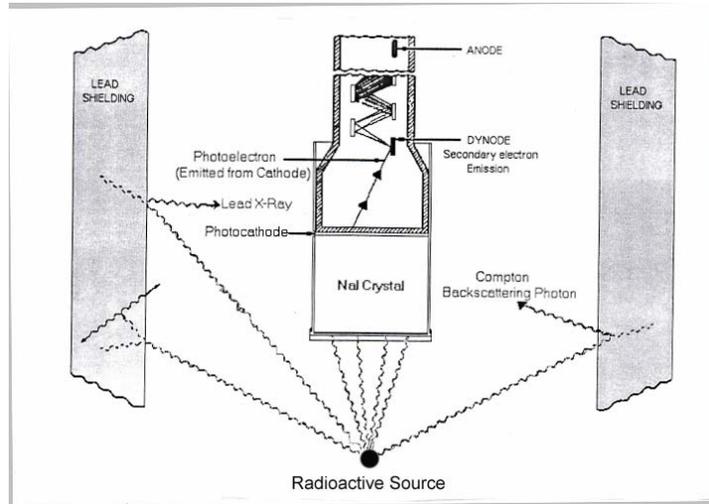


Fig 5: Detector assembly inside lead shielding—shows backscattering and Pb X-ray production.

When gamma rays transverse a scintillator material, they interact with the material by one of several mechanisms giving off visible light.

1. Photoelectric Effect: The photon is absorbed by a bound electron in an atom so that the ionized electron has kinetic energy equal to the gamma ray energy minus the electron's binding energy. This effect is most likely at energies less than ~ 150 keV and the effect is proportional to the absorber Z and the photon energy E_γ .
2. Compton Effect: The photon strikes an unbound electron or an outer electron in an atom which has a binding energy much less than the photon energy. The photon transfers part of its energy to the electron. The extent of Compton scattering in a gamma spectrum is dependent on the density of absorber material and energy of the photon. It typically manifests as a sharp edge in your spectrum, but extends to lower energies.
3. Pair Production: High energy photons may pass closely to a nucleus and transfer all of their energy into the creation of a positron/electron pair. Since the rest mass energy of an electron is 0.511 MeV, the threshold for electron-positron pair production is 1.02 MeV. Excess energy of the photon is shared equally as kinetic energy between the positron and electron. For gamma ray energies well above 1 MeV, this pair production becomes one of the most important kinds of interactions with matter.

The light emitted by the scintillator material then passes into the photocathode of the photomultiplier tube (PMT) causing the ejection of photoelectrons. See Figure 6 for a larger depiction of a PMT. These electrons in turn are then focused and accelerated on to a series of electron multiplier stages called dynodes, typically 6-10 in number.

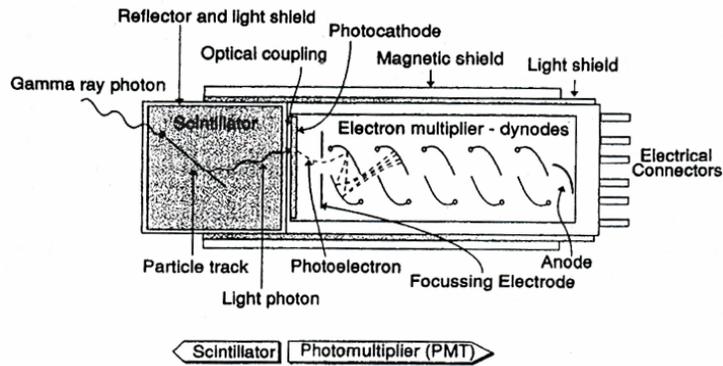


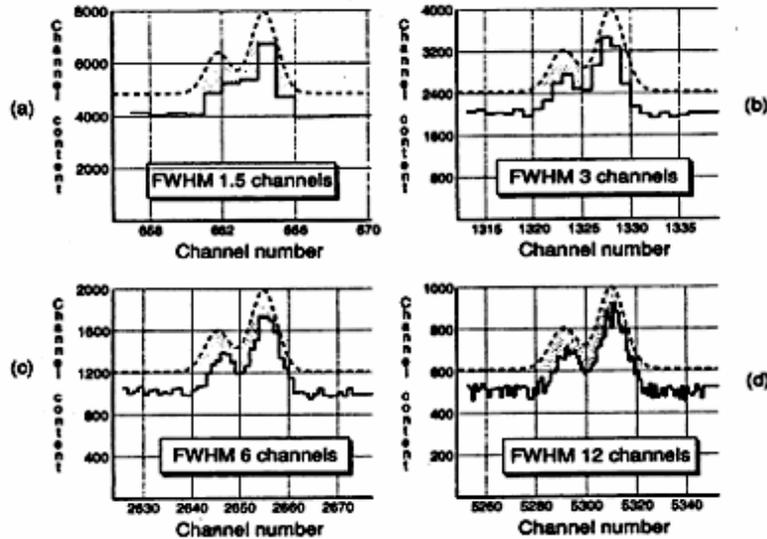
Fig 6: Photomultiplier tube assembly.

The measurable amplified signal is then collected at the anode. The amplification provided by each dynode stage is to the $\sim 10^{\text{th}}$ power, preserving the proportionality between the number of light photons ejecting photoelectrons at the photocathode and the number of electrons collected at the anode. **In other words, the charge pulse at the anode is proportional to the energy deposited in the scintillator by the incident gamma ray.** Typically, the detector-PMT is enclosed in a reflective, light-tight housing looking like an Al tube about 10-15 inches long. To minimize the presence of background gamma radiation, the detector is surrounded by lead shielding.

6) Multi-channel Analyzer (MCA) Output—Energy Resolution (FWHM) and Efficiency: The MCA displays output to the computer screen. The MCA software is ScintiVision®. The ADC and MCB are usually a computer board with input terminals on the back of the computer that accept the amp output pulses. MCB and ADC controls (number of channels, ADC offset) are controlled from the software. Figure 4 shows the effect of the ADC zero offset on the channel number - pulse height relationship. Ideally, the straight line shown in Figure 4 would go through zero. In a real laboratory however, the zero offset is adjusted to ensure that small noise pulses are not processed. Generally, the ADC zero is set no higher than 0.5% of the full spectrum number of channels. Two other controls provided by the ADC are the **lower and upper level discriminators (LLD, ULD)**. These can be adjusted to restrict the pulse heights from the amp that the ADC will accept. The LLD is usually set to block out low energy noise. The ULD is set to block pulses higher than 10 V.

One important variable that the experimenter controls is how many channels to spread the spectrum across. To understand the importance of how wide each channel should be set, see Figure 7 and suppose the two peaks have a Full Width at Half Max of 1.5 keV and are ~ 1.5 keV apart. The net peak areas are 2500 and 5000 counts on a background of 4000 counts per keV. In spectrum (a) with a selection of 1 keV per channel, the spectrum is divided into too few channels making assignment of peak energies and net peak areas difficult. Spectra (b) and (c) look OK. Spectrum (d) has 0.125 keV/channel (1.5 keV/12

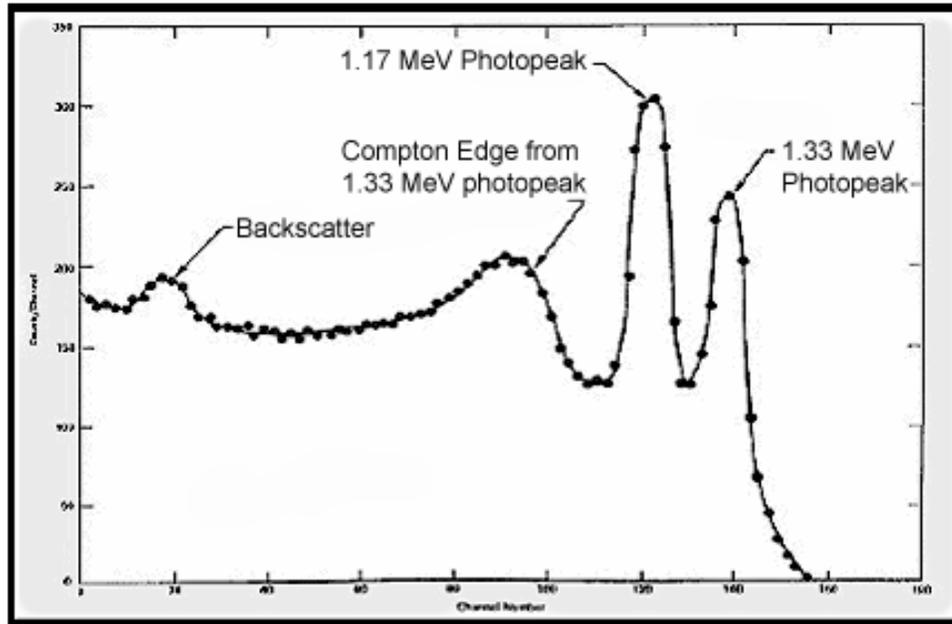
Fig 7: A demonstration of the effect of varying the number of channels used. The two peaks have FWHM = 1.5 keV, are 1 x FWHM apart, and have areas of 2500 and 5000 counts on a background of 4000 counts per keV [2], p. 87. The dotted lines represent the "real" spectrum.



channels). The problem with too many channels is that there are fewer counts per channel and therefore more uncertainty. This could be a problem with weakly radioactive samples or samples containing small peaks unless you have the luxury of counting for a very long time to generate more counts per channel. A general guideline is to start with a minimum of the FWHM = 4 channels. For a NaI detector, the energy resolution at 662 keV is approximately 5-10% with a FWHM ~ 40 keV which means using around 10 keV/channel. (Note--in the next lab you do using the solid state HpGe detector you will find that the energy resolution is considerably better). If the energy range is up to 2 MeV, about 200 channels are the minimum that could be used. In practice, 1092 or 2096 channels are usually selected to record γ -spectra with a NaI detector. Note that the number of channels selected to display a spectrum is also called the **ADC conversion gain**.

Many of the effects noted above now may become clearer to you when you look at a real gamma spectrum using the NaI detector. Figure 8, shows a spectrum of ^{60}Co which emits two photons at 1.17 MeV and 1.33 MeV. The Y axis is counts and the X axis is energy depicted by channel number. Notice the spread in the full energy photopeaks. The other features that are prominent in this spectrum is the Backscatter Peak around 250 keV. Finally, take note the Compton edge for the 1.33 MeV photopeak. This swamps effects of the lower energy photopeak.

Fig 8: NaI spectrum of ^{60}Co



PROCEDURES

Your task during the lab is to put together the individual components, provide power, connect them, and make them work. You will also look at what is involved in optimizing this type of counting system. It is very important that you should have read this procedure before the laboratory so that you can organize your data in a legible manner within the notebook. Take care to write clearly! During today's lab, draw a block diagram of your setup in your notebook, labeling all the components with manufacturer and model. Always record your instrument settings for each spectra. *Sketch* pictures of output into the notebook. Also, leave room for comment on these results you will make after you have had a chance to review your data. During this laboratory, you should create any required plots or tables and verify your observed trends (i.e. that your trend is correct) with an instructor. After you have read the material regarding NaI detectors, you may discuss the implications of your trends with the instructors. You should then go back and write your comments in the lab notebook. Remember to save all of your collected spectra (use the .SPC format) in case you want to review them later.

Note: The NaI-PMTs have been placed in a shielded enclosure with only the HV, preamp power, and preamp signal cables protruding. The HV cable is sometimes red and has extra insulation around the lead. The preamp signal cable is always black. The signal cable and HV cable connectors are different. The preamp power supply cable is grey.

Note: For today's laboratory, much of the DATA ANALYSIS section is incorporated into the PROCEDURE section. Note: Count samples long enough to collect > 1000 counts in the channel containing the maximum number of

counts (also your peak should look symmetrical such as in Figure 2 - check with TA if in doubt)!

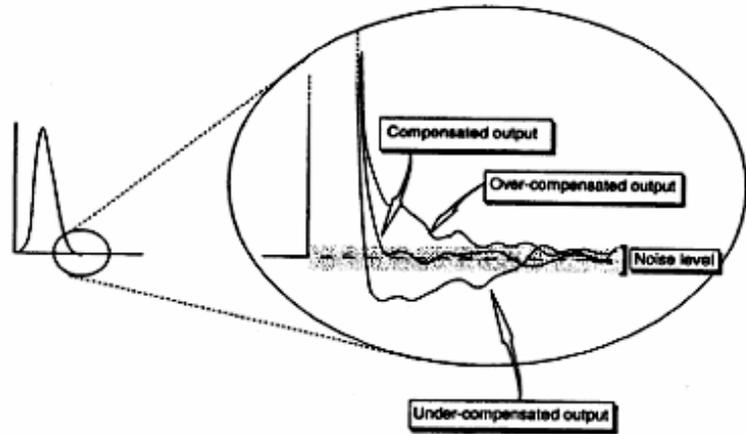
- 1) Install the HV power supply and amp into the same NIMBIN. Be sure to screw in the components securely (two screws are located on the front panel for this purpose).
- 2) Connect the detector preamp power supply (grey cable with 9-pin connector) to the 9-pin power outlet on the back of the amplifier.
- 3) Make sure that the HV supply has 0 volts (dial to zero on the HV supply) and turn off the HV switch on the front of the HV supply. Connect the HV cable from the detector to the HV power supply.
- 4) Turn the NIMBIN power to ON. Verify that the positive polarity light on the front of the HV supply is illuminated. If it is not, notify your TA. All NaI detectors require positive polarity. Have an instructor verify your settings and cable connections.
- 5) Turn on the HV, and slowly dial up to 1000 V.
- 6) Connect the preamp output to the oscilloscope (either Channel 1 or 2) using a BNC cable.
- 7) **Pre-amp Pulse Shape:** Place a γ -standard ($\sim 1 \mu\text{Ci}$, either ^{137}Cs or ^{60}Co or ^{22}Na) about 10 cm from the front of the detector. Record which standard you choose (isotope, activity, reference date). Look at the preamp output with the oscilloscope starting with sensitivity(y) $\sim 20\text{mV/division}$, and time(x) $\sim 50 \mu\text{s/division}$. You may have to adjust these settings to see the signal clearly. If you do not see a clear strong signal, you may have the trigger set too high. Sketch the output. Record the oscilloscope settings (i.e. x and y axis units). Estimate pulse heights (in mV). The preamp signal probably looks like Fig 1. If you trigger correctly, all of the signals will be superimposed on one another.
- 8) **Amp Pulse Shape:** Connect the preamp signal cable to the INPUT on the amplifier. Set the amp input to positive. Set the amp shaping time to $2 \mu\text{s}$. Connect the amp (unipolar) output to Channel 1 of the oscilloscope.
- 9) Adjust the oscilloscope settings so you can see the output from the amp clearly. Adjust the amp gain to get a $\sim 6 \text{ V}$ peak signal if you are counting ^{137}Cs or $\sim 8 \text{ V}$ if you are counting either a ^{60}Co or ^{22}Na standard. The fine gain is a multiplier for the coarse gain. So if the coarse gain is set on 200, and the fine is 1.25, the actual gain is $200 \times 1.25 = 250$. Adjust your time scale so that your amp output looks like a Gaussian peak, probably 5-10

$\mu\text{s}/\text{division}$. Record the oscilloscope settings and sketch the pulse shape. Record the x and y-axis settings and estimate the pulse height(s).

- 10) **Amp pole/zero (PZ):** If you look at Figure 1 again you can see how important it is that the amplifier pulse decays back to zero (the baseline) before processing a new pulse. If it does not, the next pulse will have a different amplitude compared to its true amplitude since the new pulse would not rise from the correct baseline. If you expand the voltage scale to 20-50 mV on the oscilloscope and observe the trailing edge of the pulses, you will see them looking like one of the situations shown in Figure 9. If your trailing pulse is either over- or under- compensated, the problem

should be corrected using a PZ cancellation circuit in the amplifier. Use a screwdriver and turn the PZ screw on the front of the amplifier: generally a clockwise adjustment returns an under-compensated output back to the baseline and vice versa for over-compensated

Fig 9: Pole-zero cancellation [2], p. 78.



output. You are trying to mimic the Compensated output situation in Figure 9. If you are lucky enough to have an automatic PZ compensation, your amplifier will have a button on the front panel instead of the screw adjustment. If this is the case, place your source on the detector such that you have ~ 1000 counts per second or higher. Push the PZ button and the amplifier automatically corrects. Your trailing edge should shift to the compensated output shown in Figure 9.

- 11) *The PZ needs to be checked each time the amp settings are changed throughout this laboratory.* After you have made your PZ adjustment, your peaks in your spectrum on the computer should exhibit no low or high energy tails (i.e. the peaks should look symmetric). You may verify this by looking at your peaks with the y-scale set on the log scale which magnifies these distortions. Connect the amp output to the ADC "INPUT" terminal at the back of the computer.
- 12) Open Scintivision [®] on your computer. In the detector window, clear any existing spectrum.

- 13) In the **Acquire** menu, select “MCB Properties”. Adjust the ADC conversion gain to 2048 channels. Start collection of the spectra. Hint: Spectra obtained with either ^{60}Co or ^{22}Na should show 2 photopeaks (look at their decay schemes!). If your spectrum does not, reduce the amp gain until the two peaks appear in the spectrum in the top quarter of memory. Save this spectrum.
- 14) Look at the amp output on the oscilloscope. Record whether all pulses appear to have the same amplitude and correlate this to the spectrum you are collecting (consider the decay scheme!).
- 15) **Amp Shaping Time:** Look at how the amp shaping time changes the display of the spectrum. Change the amp shaping time to 6 μs , check the PZ, and start collecting a new spectrum with this setting. Note any differences in the pulse shape (height and/or width) on the oscilloscope. Return the shaping time to 2 μs and readjust the PZ if necessary.
- 16) **PMT HV Dependence on Spectrum:** Look at how the peak position on the MCA and resolution change with a change in the PMT high voltage. Acquire a spectrum with the source far enough away that the dead time is less than 10%. Measure and record the peak position. Also record the amp gain settings. DO NOT change the amp gain through the next steps.
- 17) Change the PMT voltage to 1200 V (*do not go any higher!*). Check the PZ. Collect a new spectrum and save it. Record the pulse height on the oscilloscope. Note any changes in the spectrum.
- 18) Repeat with HV = 800 V. Take 2 more data points at different voltages (900 and 1100). Remember that the peak channel number is equivalent to some input voltage. By recording the peak position at the different gain settings you determine the relation between PMT gain and high voltage.
- 19) Plot the results (Channel # vs. HV) and explain the plot, particularly the behavior at the higher PMT voltage levels. Reset the HV to 1000 V and correct the PZ.
- 20) **Effect of Course Gain Setting:** From either of your ^{60}Co or ^{137}Cs spectrum, measure the peak position and calculate the count rate in a photopeak. Lower the course gain on the amp, and check the PZ. Collect another spectrum, using the same standard and shelf position, and record any changes. Save this spectrum. Calculate the count rate in the same photopeak. Comment on the results in final write-up. Change the course gain back to its previous setting.

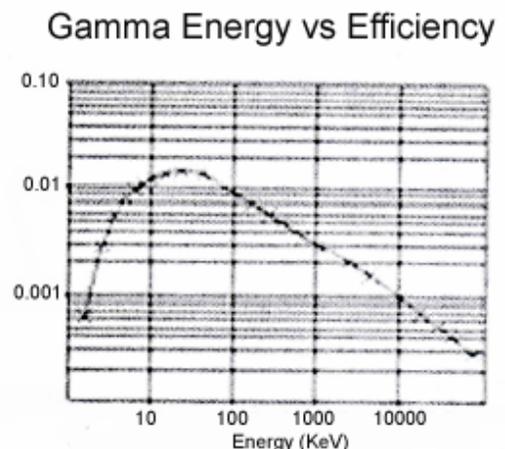
- 21) **Effect of ADC Zero Level:** Change the ADC zero level and record a new spectrum and save it. Note any changes. Change the ADC zero back.
- 22) **Effect of LLD Setting:** Change the LLD, collect a spectrum and note any changes. Save the spectrum and change the LLD back. Note: change the LLD significantly enough to see a change!
- 23) **Dead Time:** Dead time is the time when the detector system is busy processing a pulse and can not respond to new pulses. Collecting a spectrum at both high and low count rates for the same fixed time (**real time**) would result in a count rate lower than it should be being recorded for the higher count rate source. Our software can correct for this effect by providing a **live time**. The live time clock is run only when the detector system is not processing pulses. *When calculating count rates, always use the live time value.* Collect a spectrum for a minute or so with the source set up against the detector such that the dead time is > 20%. Save this spectrum.
- 24) Record the dead time, live time, and real time. Set one ROI over the entire spectrum and record the total counts. The difference between the real and live times is the time the counting system is busy processing counts. Determine how long the system is dead for each pulse it processes.
- 25) Repeat this determination with the source placed further from the detector to a point where the dead time is ~10%. Discuss in your notebook why the measured count rate would be lower than the true count rate (the rate that would be measured were there no dead time), the relationship being:

$$R_m = R / (1 + R \cdot T)$$

where T is the dead time associated with each pulse (as determined in step (24) and R is the true count rate.

- 26) **Detector Efficiency:** The NaI detector's efficiency for measuring gamma radiation is not independent of energy. When calibrating a system's performance, typically it is necessary to run several gamma standards whose gamma energies span a wide range. Then one can calculate efficiencies based on the actual dpm for the source tested and plot them vs. energy to generate an efficiency curve that may look something like Figure 10.

Figure 10



In the next lab using HpGe detectors you will perform a rigorous energy efficiency calibration so that you can measure absolute counts of environmental samples. For today, we ask that you only calculate the efficiency of one source in your sample box. Make sure that your system's settings are back to a normal state—ie HV set, amp gain set for 8 V on scope, pole/zero set, ADC controls set. Place a standard on a suitable shelf as close to the detector as possible, but having a dead time of <10%. Collect for sufficient time (~ 4000 cts in net area). Save the spectra on your memory stick and print a copy for your records.

Example Calculation:

A 1 μCi source of ^{57}Co (dated June 06) was used to acquire 4,360 counts of activity under the 136.5 KeV photopeak in 200 seconds (Live-Time) using the NaI detector. Calculate the detector efficiency at that energy.

Step 1: Calculate activity A (in dps) for the source at the time of acquisition. Using the decay equation: $A = A_0 \times \exp(-.693/t_{1/2} \times t)$ and $t_{1/2} = 271.8$ days one can calculate the absolute amount of ^{57}Co present at the time of measurement.

$$A = 1 \mu\text{Ci} \times \exp(-.693/271.8 \times 365) = 0.39 \mu\text{Ci}$$

Recall that $1 \mu\text{Ci} = 3.7 \times 10^4$ dps, therefore $0.39 \mu\text{Ci} = 14,430$ dps

Step 2: Convert the photopeak activity to dps units by dividing the count integral by the live time in seconds.

$$A_{\text{dps}} = 4360 \div 200 = 21.8 \text{ dps}$$

Step 3: Correct the measured A_{dps} for the fractional abundance of that photopeak. For the 136.5 KeV peak the fractional abundance is 0.1068.

$$\text{Total } A_{\text{dps}} = 21.8 \div 0.1068 = 204.1 \text{ dps}$$

Step 4: Calculate Detector Efficiency as follows:

$$\text{Efficiency} = (204.1 \div 14,430) \times 100 = 1.4 \%$$

POST-LAB DELIVERABLES

Your notebook should have a statement of the labs objectives and general sketches and relevant data linked to procedures.

Include the following in your final report.

- Include a brief statement of the lab's objectives.
- Include a block diagram of your counting setup including labels. In a sentence apiece describe the function of each component.
- Include a brief (1 paragraph) description of procedures. Don't get caught up in fine details here as there were a lot of things you did

today to test system performance. Just summarize.

- Include sketches of all oscilloscope traces from pre-amp and amp outputs that were asked for. You might wish to link this with the procedures and just bullet procedural tasks with outcomes (ie. sketches and comments).
- Include a summary of the effects you observed changing NIMBIN settings or the ADC settings on gamma spectra taken (including HV, Gain, ADC zero, LLD).
 - Include copies of all spectra you were asked to save (labeled)
 - Construct a graph of Channel # vs. HV (step 19) and comment.
 - Comment on why a stable HV is essential for systems used in gamma spectroscopy.
- Compare the parameters you tested for their effect on the system's dead-time. Compare the dead-time for the current setup with the dead-time (resolving time) calculated for the GM-counter.
- Compare the efficiency you measured for the NaI detector at a single energy to the general gamma efficiency you measured using the GM-counter.
- Summary statement—identify what you feel was the most significant parameter that effected your gamma spectrum and discuss why.

REFERENCES

1. Radiation Detection and Measurement, 2 ed., Glenn F. Knoll, John Wiley & Sons, Inc., 1989: For Pulse Processing and Shaping, see pp. 555-583; Pulse Height Analysis, see pp. 584-681.
2. Practical Gamma-Ray Spectrometry, Gordon Gilmore and John Hemingway, John Wiley & Sons, Inc., 1995.
3. Parts of this laboratory handout were based on the Gamma-Ray Laboratory, part of an "Advanced Physics Lab, PHY 4803L" course at the U. of Florida.

(NCSS Lab 5)

Gamma Ray Spectroscopy with Semiconductor Germanium Detectors

OBJECTIVES

The objectives of today's lab are three-fold:

- Assemble a HPGe detector counting system.
- Characterize basic operational properties of HPGe (including Energy-Channel, Energy resolution, Detector Efficiency and Peak-to-Total Ratio).
- Analyze environmental samples to determine the nature of the radioactive constituents within them.

INTRODUCTION

The high-purity germanium (HPGe) detector is really a solid state analog of the gas filled ion chamber (GM tube) used in the GM experiments. However, there are some differences. Germanium's density is much higher (5.33 g/cm^3 vs. $2 \times 10^3 \text{ g/cm}^3$ comparing GM gas vs. Ge crystal). The charge carriers in the germanium crystal are electrons and holes vs ion pairs in a GM tube. The charge carriers in a HPGe detector travel through a nearly perfect single crystal rather than through a gas. The mobilities of the electrons/holes are much faster in germanium, $\sim 10^1 - 10^5$ times faster than the ion pairs in a GM tube. The energy required to produce an electron/hole pair is ten times lower, 2.96 eV vs. 30 eV for an ion pair formation in a GM tube or 170 eV for the electron/hole pair in a NaI(Tl) detector. Finally, the number of electron/hole pairs produced is directly proportional to the energy deposited by the gamma or x-ray. Recall that there is no energy correlation in the GM tube.

The principle advantage of using an HPGe detector in place of the NaI(Tl) detector is enhanced energy resolution (see Attachment 1). The typical energy resolution that could be obtained with NaI(Tl) is $\sim 7\%$ for the 0.661 MeV ^{137}Cs γ -ray while the energy resolution for a HPGe detector is typically $\sim 0.2\%$. As a result, spectra containing many gamma ray peaks are much better resolved using a HPGe detector compared to a NaI(Tl) detector. Figure 1 shows a comparison of the NaI(Tl) spectrum with an HPGe spectrum of ^{166}Ho and speaks for itself.

On the downside, the Z (32) for Germanium is much lower than I (53) in the NaI detector. Recall how the probability of a gamma ray interacting with a material is proportional to Z. The detector crystal size is much smaller for an "affordable" HPGe detector compared to a NaI(Tl) detector. Both of these factors result in a much lower *detection* efficiency for HPGe detectors, $\sim 0.1\%$, compared to 10-60% for NaI(Tl) detectors.

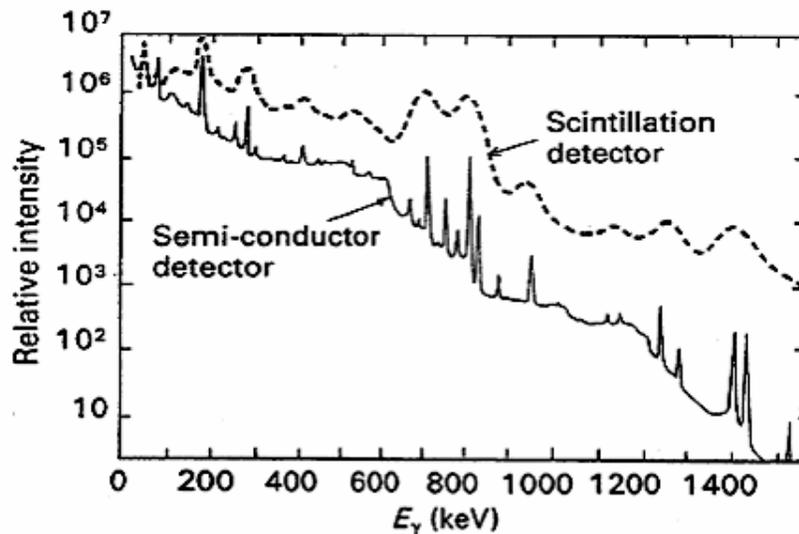


Fig. 1: A comparison of the NaI(Tl) spectrum and an HPGe spectrum of ^{166m}Ho .

Also, HPGe detectors are much more expensive and require liquid nitrogen operating conditions for reasons explained below. Even though the Ge detector crystal is only the size of a walnut, the cooling system takes up a large footprint in the lab as is shown in Figure 2. The Dewars used in this laboratory are ~ 30 L in volume. Smaller dewars are available for “portable” systems. Still, the advantages outweigh the disadvantages in many applications.



Fig. 2: Different types of dewars used to cool HPGe detectors (picture provided by Canberra, Inc.)

SEMICONDUCTOR DETECTORS

Detectors based on semiconductors primarily employ germanium to detect gamma and x-rays. The electronic band structure of insulators, metals conductors and semiconductors are shown in Figure 3. The difference between these materials is in their electronic structures. See also E&V, pp. 229 - 232. In summary, the energy gap between the valence band and the conduction band is the band gap, E_g . In an insulator E_g is ~10 eV, but in a semiconductor it is much smaller, ~ 1 eV. In both materials, the valence band is full. The small band gap in a semiconductor allows electrons to easily be promoted to the conduction band even by thermal excitation. In an absolutely pure semiconductor, the number of electrons would be matched by an equal number of holes. Such a material is described as intrinsic and many germanium

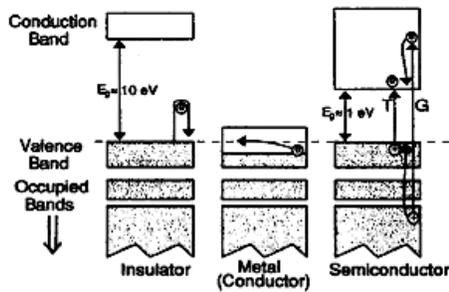


Fig. 3: Schematic diagram of the electronic band structure in insulators, metals, and semiconductors

impurities). The presence of these impurities acts just like the TI impurity in NaI crystals by introducing new energy levels either just above (p-type) or just below (n-type) the conduction band. The semiconductor gamma ray detector arises when one places a piece of n-type material next to a p-type material (Figure 4a). Figure 4 shows the interactions. At the junction, some of the donor impurities cross over the junction to cancel with impurities on the other side. This creates a depletion region surrounding the junction (Figure 4b). Also note that the movement of these impurities creates a space charge in the region which generates a voltage across the junction, typically 0.4 V in germanium (Figure 4c,d). This depletion region is the active region of the detector. We would wish to have as large a depletion region as possible to maximize our detector volume and efficiency. This is done by applying positive voltage to the n side of the junction. The width of the depletion region (d) is determined by the following relationship:

$$d \sim [2 \kappa \mu \rho (V_o + V_b)]^{1/2}$$

where V_o and V_b are the contact and bias voltages, κ is the dielectric constant, ρ is the resistivity of the detector material (germanium) and μ is the mobility of the majority charge carrier in the material. We can conclude that the value of d

detectors are referred to as intrinsic detectors. It is actually not possible to manufacture a crystal completely free of impurities. All crystals have a predominance of either electron rich impurities (n-type, where 'n' is for negative donor impurities) or electron poor impurities (p-type, where 'p' is for positive acceptor

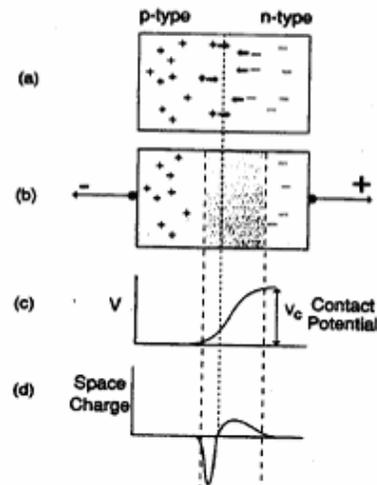


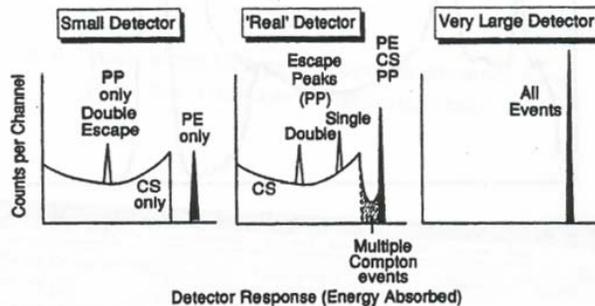
Fig. 4:(a) p-n junction before charge carrier redistribution; (b) depletion region created by carrier redistribution; (c) variation in potential across junction; (d) variation in space charge across junction [1], p. 43.

increases with increasing bias voltage (operating voltages are typically between 2000 and 4000 V) and material resistivity. ρ is proportional to the inverse of the impurity concentration. Therefore, germanium crystals are manufactured to be as intrinsic as possible (adding significantly to their cost!).

The interaction of the γ ray with the Ge crystal promotes electrons to the conductive band leaving behind holes in the valence band. The applied voltage across the detector (bias voltage) sweeps these charges from the crystal to where they are accumulated in the preamp and converted to voltage pulses. Due to the small band gap in germanium, all HPGe detectors are kept cooled to $\sim 77^\circ$ Kelvin using liquid N_2 . This reduces the background current due to thermal excitation of electrons. Fortunately, the electronics to convert these charge pulses to a histogram are identical to those used with many other detectors.

Positron Annihilation: Except for the 0.511 MeV annihilation gammas, most gamma rays have very small natural line widths compared to the detector (uncertainty in total number of charge carriers) and electronic (noise) line widths. With positron annihilation however, there is an additional contribution to the line width. Positrons slow down in a material to the point where they annihilate with electrons producing two gamma rays, each with 0.511 MeV and traveling in opposite direction from each other. Without going into detailed physics, these photons are Doppler shifted because the original electron is not at rest. This introduces an additional uncertainty in the energy of the photons resulting in larger FWHM compared to other gamma rays. In addition, single and double escape peaks which result from the escape of one of the annihilation photons from the detector will also have larger FWHMs. Figure 5 shows how escape peaks will manifest artifacts on your spectrum.

Fig 5: Single and double escape peaks.



Remember—do not to use the 0.511 MeV peaks in either your energy or efficiency calibrations.

Environmental Radioactivity: There are many radioactive isotopes in our environment. Some are manufactured purposefully using reactors and accelerators for nuclear medicine and industrial applications. Unintentional radioisotopes are found due to the wide scale testing of nuclear weapons that ended in 1963. Some isotopes occur naturally having been formed either by stellar evolution or through production by cosmic rays. Examples of these are ^{40}K ($t_{1/2} = 1.3 \times 10^9$ year), ^{232}Th ($t_{1/2} = 1.4 \times 10^{10}$ year), ^{235}U ($t_{1/2} = 7.0 \times 10^8$ year), ^{238}U ($t_{1/2} = 4.5 \times 10^9$ year), and ^{14}C ($t_{1/2} = 5730$ year). Other isotopes occur naturally

because they are part of a decay chain with ^{232}Th , ^{235}U or ^{238}U as the parent. The sequence of daughters are found in Ref. 2.

APPARATUS

1. A gamma ray spectrometer with a HPGe solid state detector cooled with liquid nitrogen.
2. High voltage supply up to 5000 volts, spectroscopy grade amplifier, oscilloscope, MCA
3. Sample mount with fixed shelf positions attached to the detector unit.
4. A set of radioactive γ -ray standards (See Appendices 4 and 6).
5. Gamma source kit for calibration.
6. Environmental samples.

PROCEDURE

The purpose of this experiment is to study some of the properties of HPGe detector systems and to use your detector system to analyze some materials that would be impossible to analyze with any other type of detector. You will put together and characterize a HPGe detector system in the same manner as the NaI(Tl) detectors. **Many of the procedures are identical to the previous lab.** You will also be taking a look at some interesting examples of radioactivity in our environment. One type of material you will study is called monazite. Monazite is a sand-like material that contains rare earth chlorides including uranium and thorium. It is found in India, Nigeria, and in New Mexico. Another material is the infamous pitchblende that Madame Curie worked with to isolate Polonium. You will also take a look at background radiation in the counting room. We will also have on hand samples of Fiesta ware ceramic tableware, uranium glass from the Depression era and a soil/sand sample from a local beach.

1. Install the HV power supply and amplifier into the same NIMBIN. Connect The detector preamp power supply to the power outlet on the amplifier. Connect the HV supply to the detector. Set the HV supply voltage to zero volts and turn off the on/off switch. Turn the NIMBIN power to ON. Adjust the HV polarity to match the detector requirements (the voltage and polarity are stamped on the outside of the aluminum can of the detector). Have a TA verify your settings. Slowly dial up the voltage.

2. Connect the output from the preamplifier to a spectroscopy grade amplifier. The shaping time constant should be set to 6 microseconds for the best energy resolution.
3. Place a standard source ~ 10 cm from the HpGe detector.
4. Connect the unipolar output of the amplifier to the oscilloscope.
5. Adjust amplifier gain for a ^{22}Na or ^{60}Co standard so that the size of the pulses are ~8 volts. Adjust the pole-zero of the amplifier for any under-shoots or over-shoots of the pulse shape on the oscilloscope so as to achieve optimum resolution.
6. **System Calibration**: Now you are ready to measure spectra of the γ -ray standards. Adjust the amplifier gain so that all γ -rays of interest, ranging from 60 keV to 1.33 MeV are present in a single **display of 4096 channels**. Make sure that your detector settings used are suitable for measuring the the full range of gamma energies from the standards in your source kit which includes ^{57}Co , ^{133}Ba , ^{109}Cd , ^{54}Mn , ^{137}Cs , and ^{22}Na or ^{60}Co . That is, not only should the 1.27 MeV γ -ray of ^{22}Na or the 1.32 MeV γ -ray of ^{60}Co be in the top quarter of the memory but the 122 keV γ -ray peak of ^{57}Co or the 88 keV γ -ray peak of ^{109}Cd should be at a channel about one-tenth that of the 1.27 MeV γ ray peak. Essentially you want to bracket both low and high energy gammas when constructing and energy calibration curve. Use the **same** source that you used in the NaI(Tl) lab to calculate detector efficiency as later in the final write-up you will be asked to comment on differences. Also remember that two of your sources must include ^{57}Co and ^{60}Co . Select a shelf as close to the detector as possible, but also having the Dead Time < 10%. Collect for sufficient time (i.e. 4000 counts in net area). Print out spectra for each standard and record the following for each γ -ray photopeak to be used in calibrations in Table 1:
 - a. Peak channel number.
 - b. Integrated peak counts with background subtracted.
 - c. Live-Time
 - d. Full width at half maximum, (FWHM), in energy units.

Energy Calibration: Recall that the size of the output amplifier signal is directly proportional to the gamma ray energy deposited in the detector and the way it interacts with the detector. Electronic factors also influence the size of the pulse, i.e. amplification provided by the amp and preamp gain and bias HV setting applied to the solid-state detector. The **energy calibration** tells you which channel corresponds to which energy—this is a critical calibration when you wish to use your system to interrogate “unknown” environmental samples. To perform the calibration, one measures the spectrum or spectra of radioactive sources which emit

gamma rays at precisely known energies. The channel number, C_γ , for the center of the photo-peak is recorded and plotted vs. the E_γ . Once the calibration is known, energies of unknown peaks may be assigned. **The energy calibration is specific to a chosen set of hardware settings. Change the amplifier gain and your previous energy calibration is no longer valid!**

Efficiency Calibration: The amount of activity of a sample may be directly calculated from a net peak area determination if one has performed an efficiency calibration on the detector system. There are several efficiencies associated with these detectors. Your calibrations will determine the **absolute full-energy peak efficiency** which relates the peak area to the number of gamma-rays emitted by the source and will be referred to as efficiency from now on. Calculating efficiency (ϵ) is straightforward since it is simply a ratio of the number of counts (subtracting background) detected in a particular peak (R) related to the emission rate of the source:

$$\epsilon(E) = R / (A_S \times f_i)$$

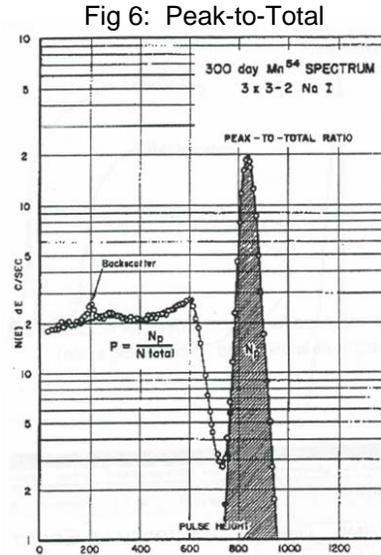
where A_S is the emission rate in disintegrations per second. Typically, a source activity and a reference date is stamped on the calibration source and the count rate is decayed to this reference date in order to use the equation. Here f_i is the fraction of disintegrations that emit the gamma ray at energy E (recall the example given to you in the previous PAH lab). The **fractional abundance** of the radionuclide can be determined from the nuclide's decay scheme. For example, ^{60}Co decays by γ -ray emission to ^{60}Ni , 99% of the time to an excited state that is 2.505 MeV above the ground state, and 1% of the time to the ground state. This state rapidly decays by emission of a 1.173 MeV gamma ray followed rapidly (ps) by a 1.332 MeV gamma ray. Thus each gamma ray has a fractional abundance of 0.99. Values for fractional abundance are available in your Laboratory Manual Appendix 4 and 6. Note that standards are provided with activities as of a reference date. The user must decay correct from the reference date to the measurement date before calculating the efficiency.

Like the NaI detector, the HpGe detector efficiencies will not be independent of energy. It is most practical if one chooses standards for calibrating the system whose gamma rays span a wide energy range. Then one may calculate efficiencies and plot them vs. gamma ray energy to generate an efficiency curve. In this manner, unknown activities of radionuclides may then be calculated from their net peak areas (R) by rearranging the efficiency equation:

$$A_S = R / [f_i \times \epsilon(E)]$$

where $\varepsilon(E)$ is interpolated from the efficiency curve and A_S is in dps.

Other parameters that directly relate to the detector photopeak efficiency is the **Peak-to-Total Ratio** and **Peak-to-Compton Ratio**. These ratios are defined as the fraction of the total number of events in the spectrum which appear in the photopeak relative to either the total spectrum's counts or the counts under the Compton plateau. The higher this fraction is, the higher the efficiency. Figure 6 shows an example of a Peak-to-Total calculation.



7. **Environmental Samples:** Pick two samples from the collection of environmental samples (do one of the ore samples plus one of the other samples) and acquire spectra on each similar to that of the calibration spectra. Set collection times sufficient to collect ~ 4000 counts in primary peaks. Save the spectrum to your memory sticks for later analysis as well as print a hardcopy. For environment samples, you should place them as close to the detector as possible though keep the dead time less than 5%.
8. **Background:** When everyone has finished collecting sample spectra and put all of the sources behind shielding, collect a background spectrum of the same length of time you used for your environmental samples.
9. When finished collect hardcopies of all your spectra, dial down the HV to zero, wait one minute, and then turn off the power to the Nimbin. **Do not disconnect electronics as you will be using this system in the next lab on Secular Equilibrium.**

DATA ANALYSIS

1. Construct a plot of Gamma Energy vs. Channel Number for energy calibration and compute KeV per channel (refer to Appendices 4 and 6 in the Lab Manual for information of gamma energies—or check out the wall chart in the counting room).
2. **Energy Resolution:** Calculate energy resolutions from your gamma standards (equal to Photopeak FWHM ÷ Energy) and plot % Resolution vs. Gamma Energy. Use GammaVision® to calculate FWHM values in energy units.

3. **Detector Efficiency:** Calculate the detector efficiency for each photopeak you integrated using your gamma standards and record results in Table 1. Refer to Appendix 6 at the back of this manual for the decay branching ratios of the different photopeaks. Refer back to the example shown in the NaI(Tl) lab for guidance on how to do these calculations. Make a plot of Detector % Efficiency vs. Gamma Energy.
4. **Peak-to-Compton Ratio:** From either your ^{60}Co or ^{22}Na spectrum, calculate the Peak-to-Compton ratio: The Peak-to-Compton ratio is used to characterize an HPGe detector's performance and is a number provided by the detector manufacturer (record this value off the toe tag on your detector for comparisons). It is the ratio of the largest number of counts in the 1332.5 keV peak (^{60}Co spectrum) or 1274 keV peak (^{22}Na spectrum) to the average channel count in the Compton continuum between 1040 and 1096 keV (^{60}Co spectrum) or between 828 and 884 keV (^{22}Na spectrum). Note: The energy region does not have to be exactly the energies specified here but the region should encompass the relatively flat Compton continuum before the photopeak. To obtain the average channel count in the Compton continuum, create a ROI to determine the integral area of the region and divide by the number of channels in the region.
5. **Peak-to-Total Ratios:** For your calibration spectra that contain only one photopeak, calculate Peak-to-Total Ratios and record data in Table 1. Construct a plot of the Peak-to-Total Ratios vs. Gamma Energy.
6. **Environmental Samples:** You will be analyzing two samples. In the first part, we just want you to identify the gamma peaks from a single sample your team selects from the box of ores and commercial goodies. The spectrum will be complicated with several photopeaks to identify. You must consider the contributions from background radiation to your sample spectrum and omit those peaks from the spectrum. You can do this using the GammaVision software. This year, depending on timing, I may have some environmental research samples for you to analyze. These samples come from a coring project in a river delta located in China. Likely, you will be looking at a single radionuclide (Pb-210) and quantifying its amount. To do this you will have to rely on your system's energy efficiency calibration and also correct for background radiation. Each team will have a different sample from the core and at the end of the lab all teams will compile their data to reflect on trends. I will provide you with background information on the collection site. Also, these samples are likely to be extremely low in counts. Therefore, you will have to set these up for overnight counting and retrieve the data the next morning before class.

POST-LAB DELIVERABLES

Notebook

- Include a brief statement of the lab's objectives.
- Include a block diagram of your HPGe system's components.
- Include of brief description of the procedures.
- Include copies of all your gamma spectra including calibration spectra and label all relevant parts.
- Include a completed copy of Table 1.
- Include any observations from the lab that may have impacted your data.

Final Report

- Include a summary of what this lab was about (key elements we are looking for include title, date, lab partners, statement of objectives a statement on how these objectives were met through a description of the basic principles of the HPGe system.
- Include plots of the following:
 1. Energy vs. Channel Number
 2. Resolution vs. Energy
 3. Peak-to-Total vs. Energy
 4. Efficiency vs. Energy
- Include sample calculations of Energy Resolution, Peak-to-Total resolution and Peak-to-Compton resolution. If the Peak-to-Compton value is posted on your detector compare your measured value to that of the manufacturer.
- Include a discussion of the performance of your HPGe system by comparing the trends you observed in the above plots with those you obtained from the NaI(Tl) scintillation detector. How do the values for Energy Resolution and Detector Efficiency compare for a fixed energy? Discuss the trends you observed in your energy-efficiency plot. Why would you expect efficiency to decline with increasing gamma energy?
- Include gamma spectra of your environmental samples and background spectra with a discussion on what components you identified in each sample considering decay chains of fission products (ex. consider the thorium decay series). **Remember to label x axis in energy units—not channel number.**

- Discuss how the basic principles of operation of the HPGe detector are similar and different from the NaI detector.
- Discuss what you identified in your first environmental sample from the box of ores and commercial products. How much of the background spectrum was similar to your sample spectrum? Why would you expect to see similarities? What might be a primary source of the background radiation in the counting room (hint—the walls are thick concrete).
- Discuss any trends you observed from the core samples looking at depth of penetration and nuclide concentration.
- Include a summary statement describing the merits and pitfalls of HPGe spectroscopy. In what situations would an HPGe detector be ideal? When is a NaI(Tl) detector better suited to the task?

REFERENCES

1. Practical Gamma-Ray Spectrometry, Gordon Gilmore and John Hemingway, John Wiley & Sons, Inc., 1995.
2. Natural Decay Series:
<http://www.ead.anl.gov/pub/doc/NaturalDecaySeries.pdf>
3. Naturally Occurring Radioactivity:
<http://www.physics.isu.edu/radinf/natural.htm>
4. For further reading on HPGe detectors, you may consult:
Radiation Detection and Measurement, 2 ed., Glenn F. Knoll, nd, John Wiley & Sons, Inc., 1989.

Table 1.

Calibration Data

Photopeak Energy (KeV)	Channel Number	Integrated Counts	Live Time (sec)	FWHM (KeV)	Resolution (%)	Detector Efficiency (%)	Peak-to-Total Ratio

Note: Peak-to-Total Ratios need only be listed for those sources you used that had a single photopeak.

Secular Equilibrium in Radioactive Parent-Daughter Decay

OBJECTIVES

1. To demonstrate the basic principles of secular equilibrium using ^{113}Sn decay to its radioactive $^{113\text{m}}\text{In}$ daughter.
2. To build and test a radioisotope generator using ^{113}Sn on an anion exchange column.

INTRODUCTION

Chromatographic methods, of which ion exchange is only one of many variations on a theme, are separation methods. A chromatographic method is one in which two immiscible phases are brought into contact: one phase is stationary while the other moves. The stationary phase, which is usually packed into a column, can be a solid or a liquid coated onto a solid support. The mobile phase can be a gas or liquid. Separation is accomplished as follows: (see Figure 1). The sample to be separated (fractionated) is dissolved into the mobile phase and placed on the column of stationary phase. The sample, carried by the gravity induced flow of the mobile phase, begins to migrate through the stationary phase (panel 1). Since each of the components that comprise the mixture are different in their chemical nature, each will interact to a different degree with the stationary phase. The degree of interaction defines the migration rate: hence little interaction is exhibited by a fast migration rate. The individual components separate, due to their different migration rates (panels 2 and 3). If the mobile phase exiting the column (the eluent) is collected in fractions, the components of the mixture can be isolated. Figure 2 demonstrates the power of this method to separate multi-component samples.

The numerous modifications on the basic chromatographic format are usually classified based on the nature of the interaction between the stationary phase and the components to be separated: Therefore, major classifications of chromatography include:

Adsorption: The analytes adsorb onto the stationary phase due to interactions with surface functional groups. This type of chromatography occurs naturally in soils, and is used extensively in large scale water treatment.

Partition: When the stationary phase is a viscous liquid coated on to a solid support, separations based on polarity differences can be used. If a nonpolar liquid is used, polar components will pass through quickly, while nonpolar ones will not. Partition chromatography has been widely applied in the biological sciences.

Gel: This type of chromatography, used exclusively by biochemists, separates based on size differences of the components. The stationary phase, called a gel, is carefully synthesized so as to have accurately known pore dimensions. Large molecules, which

cannot fit into the pores, pass through quickly, while the smaller ones are slowed by the diffusion into and out of the pores.

Ion-exchange: No matter what the type, each is just a variation on the basic chromatographic process introduced above. The last major group of chromatographic methods, those based on ion exchange, will be the focus of this experiment. In ion exchange chromatography, separation is based on the interaction of solute ions with fixed charged sites on the stationary phase. These stationary phases are prepared as follows: A polystyrene resin is prepared by the copolymerization of styrene (A) and divinylbenzene (B) (Figure 3). This results in a resin with the repeating unit structure shown in Figure 4. Certain positions of this matrix can be chemically modified to contain the ionic groups needed for chromatography.

CATION EXCHANGE PROCESS

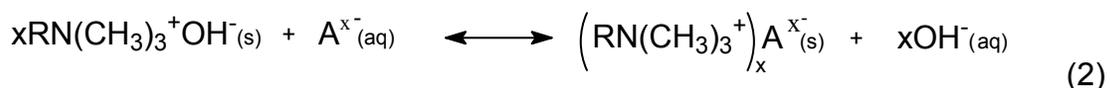
If the individual ionic sites are sulfonic acid groups (Figure 5), the resin is a cation exchange resin. In a highly acidic medium, all the ionic positions will be occupied by the hydrogen ion based on an electrostatic attraction. The hydrogen ions, however, can be displaced if another ion more desirable comes along (see Figure 6). The following equation illustrates a typical cation exchange process:



where M^{x+} represents a specific cation in solution, and $(\text{RSO}_3^-)_x\text{H}^+$ is a sulfonic acid commonly used as a functional group on the solid support. For separation to occur, the cation in solution must have a greater attraction for the sulfonic ion (RSO_3^-) than the hydrogen ion (H^+). Eventually the mass action of hydrogen ions in solution "pushes" the cations through the column. As the solution moves down, the cations will have detached and re-attached to numerous functional groups along the way. Also note that the resin as supplied by the manufacturer may be provided with a different counter ion than H^+ (that resin is said to be in the hydrogen form). Another common form is the Na form and the counter ion in that case is, naturally, Na^+ .

ANION EXCHANGE PROCESS

The following equation illustrates a typical anion exchange process:



where A^{x-} is the anion in solution and $x\text{RN}(\text{CH}_3)_3^+\text{OH}^-$ is an amine commonly used as a

functional group on the solid support. As with the cation exchange process, for separation to occur the anion needs to have a greater attraction for the amine ion $x\text{RN}(\text{CH}_3)_3^+$ than does the hydroxyl ion (OH^-). The mass action of numerous hydroxyl ions in solution eventually "pushes" the anions through the column. Another common form that this resin is provided in is the chloride form with the counter ion being Cl^- .

LAW OF MASS ACTION

Using the anion exchange process as an example, the application of mass action leads to the following equation:

$$K = \frac{\left[\left(\text{RN}(\text{CH}_3)_3^+ \right)_x \text{A}_{(s)}^{x-} \right] \left[\text{OH}_{(aq)}^- \right]^x}{\left[x\text{RN}(\text{CH}_3)_3^+ \text{OH}_{(s)}^- \right] \left[\text{A}_{(aq)}^{x-} \right]} \quad (3)$$

where K is the equilibrium constant and the brackets represent the molar concentrations.

When one of the ions predominates in both the liquid and solid phases, such as the hydroxyl ion in the anion exchange process, then equation (3) can be simplified because:

$$\left[\text{OH}_{(aq)}^- \right] = \left[x\text{RN}(\text{CH}_3)_3^+ \text{OH}_{(s)}^- \right] \quad (5)$$

and,

$$K_d = \frac{\left[\left(\text{RN}(\text{CH}_3)_3^+ \right)_x \text{A}_{(s)}^{x-} \right]}{\left[\text{A}_{(aq)}^{x-} \right]} \quad (6)$$

where K_d is the distribution coefficient of the specific observed anion between the solid and liquid phases. A high value for K_d (typically > 50) indicates a high affinity of the ion to resin. A low K_d value (< 5) indicates a poor binding affinity to the resin

RADIONUCLIDE GENERATORS

Radionuclide **generators** are often used to store short-lived nuclides until they are needed (generally for nuclear medicine applications). A generator is a device containing a parent-daughter decay chain of at least 3 isotopes in which the parent decays through a

daughter to a stable or very long lived isotope. **The daughter is a different element from that of the parent, and, hence, can be chemically separated from the parent.** Many generator systems use chromatography to affect the chemical separation. The parent isotope is essentially irreversibly adsorbed on a column and the daughter is easily eluted with the appropriate agent. The most frequently used generator produces 6.0-hr ^{99m}Tc for medical diagnosis. The parent nuclide is 67-hr ^{99}Mo (decay scheme shown in Figure 7). Table 1 below lists several other useful generator systems.

Table 1

Mother nuclide	Decay properties	Daughter nuclide	Decay properties	Application
^{44}Ti	EC, γ ; 47.3 y	^{44}Sc	$1.5 \beta^+$; 1.16 γ ; 3.93 h	Teaching
^{68}Ge	EC; 270.8 d	^{68}Ga	$1.9 \beta^+$; 1.08 γ ; 1.135 h	Medical
^{87}Y	EC; 3.35 d	^{87m}Sr	0.39 γ ; 2.80 h	Medical & teaching
^{90}Sr	$0.5 \beta^-$; 28.5 y	^{90}Y	$2.3 \beta^-$; 2.671 d	Heat source [†] , Calibration source
^{99}Mo	β^- , γ ; 65.9 h	^{99m}Tc	0.14 γ ; 6.0 h	Medical
^{113}Sn	EC, γ ; 115.1 d	^{113m}In	0.39 γ ; 1.658 h	Medical
^{132}Te	β^- , γ ; 78.2 h	^{132}I	$2.1 \beta^-$, γ ; 2.28 h	Medical
^{137}Cs	β^- , γ ; 30.0 y	^{137m}Ba	0.66 γ ; 2.55 m	Gamma radiography, Radiation sterilization [†]
^{140}Ba	β^- , γ ; 12.75 d	^{140}La	β^- , γ ; 1.678 d	Lanthanum tracer
^{144}Ce	β^- , γ ; 284.9 d	^{144}Pr	$3.0 \beta^-$; 17.28 m	Calibration source
^{210}Pb	β^- , γ ; 22.3 y	^{210}Bi	$1.2 \beta^-$; 5.01 d	Calibration source
^{226}Ra	α ; 1600 y	^{222}Rn	α ; 3.825 d	Medical
^{238}U	α ; 4.468×10^9 y	^{234}Th	β^- , γ ; 24.1 d	Thorium tracer

In-Sn GENERATOR

For this lab, you will prepare a generator column using the ^{113}Sn - ^{113m}In parent-daughter pair. The decay scheme is shown in Figure 8. The ^{113}Sn parent is long-lived ($t_{1/2} = 115.1\text{d}$) and the ^{113m}In daughter is short-lived (99.5 min). A commercial anion-exchange resin is utilized in this experiment: Dowex 1X-8, 100-200 mesh size to separate Sn from In. This resin strongly binds Sn^{+4} in all concentrations of hydrochloric acid (HCl) $\geq 4 \text{ N}$ with $K_d \sim 1000$. The ^{113m}In that grows in as a result of the ^{113}Sn decay can be eluted from the column with H_2O since Indium is not retained by the resin in H_2O , $K_d < 1$. The ^{113}Sn parent activity may then be eluted from the column with nitric acid, (HNO_3) since Sn is not retained by this resin in any normality of HNO_3 .

Some terminology commonly used when defining generator performance include: (i) **Breakthrough** which is the fraction of parent isotope eluted with the daughter isotope; and (ii) **Elution Yield** which is the fraction of daughter isotope recovered in its elution from the column divided by the amount of daughter isotope loaded on the column. In Nuclear Medicine application of generators, breakthrough is a more important concern as it can mean higher radiation exposures to patients.

PRINCIPLES OF SECULAR EQUILIBRIUM

The generator column may be used to examine secular equilibrium that the $^{113}\text{Sn}/^{113\text{m}}\text{In}$ pair exhibit. **Radioactive equilibrium exists when a radioactive nuclide is decaying at the same rate at which it is being produced.** In secular equilibrium, the decay rates of all radionuclides within a series are nearly equal, i.e. all of the daughters appear to have the half-life of the parent. Two conditions are necessary for secular equilibrium. First, the parent radionuclide must have a half-life much longer than that of any other radionuclide in the series. Second, a sufficiently long period of time must have elapsed, for example ten half-lives of the decay product having the longest half-life, to allow for ingrowth of the decay products. The number of daughter nuclei present at any time "t" is proportional to the initial number of parent and daughter nuclei present, N_1^0 , and N_2^0 , respectively, and the ratios of the two decay constants, parent (λ_1) to daughter (λ_2):

$$N_2(t) = \left(\frac{\lambda_1}{\lambda_2 - \lambda_1} \right) N_1^0 \left(e^{-\lambda_1 t} - e^{-\lambda_2 t} \right) + N_2^0 e^{-\lambda_2 t} \quad (7)$$

This equation incorporates the fact that the daughter grows in at the rate at which the parent decays and the daughter decays at her rate of decay. Using the $^{113}\text{Sn}/^{113\text{m}}\text{In}$ system as an example, if we use a chromatography column to separate the two elements, at $t=0$ one fraction (the resin on the column) will have pure ^{113}Sn and one fraction (the elution volume) will have pure $^{113\text{m}}\text{In}$. The activity curve of the pure $^{113\text{m}}\text{In}$ fraction will then give a simple exponential decay based on the daughter half-life (see closed diamond curve in Figure 9). The activity curve for the initially pure ^{113}Sn fraction is based on the first term in Equation (7) since N_2^0 is equal to zero. However after some time has elapsed, it is possible to detect the presence of $^{113\text{m}}\text{In}$. In fact, the number of $^{113\text{m}}\text{In}$ atoms increases with its half-life. Therefore, after one half-life of $^{113\text{m}}\text{In}$, 50% of the maximum number of atoms possible has been created (open circle curve in Figure 9). The maximum value of $^{113\text{m}}\text{In}$ (at $t = \infty$) is given by:

$$N_2 \lambda_2 = N_1 \lambda_1 \quad (8)$$

which indicates that over time, the number of $^{113\text{m}}\text{In}$ atoms becomes constant. At that time, the rate of decay of $^{113\text{m}}\text{In}$ is equal to the rate of decay of ^{113}Sn (which defines secular equilibrium). Remember though, that the actual number of atoms of $^{113\text{m}}\text{In}$ is much less than the number of atoms of ^{113}Sn . Due to the much longer half-life of ^{113}Sn , no reduction in activity of the parent (closed circles in Figure 9) can be observed over the time of measurement.

DECAY CURVE ANALYSIS

An examination of the decay scheme (Figure 8) of ^{113}Sn shows that it decays by electron capture (EC) primarily to $^{113\text{m}}\text{In}$ (98%) and also to higher energy states at 647 keV and 1.0297 MeV (not included in the figure) above the ground state (1.7%, 0.015% respectively). The $^{113\text{m}}\text{In}$ 1.658 hour isomeric state at $E = 392$ keV above ground decays via the emission of a 392 keV γ -ray. The 392 γ -ray energy and half-life determination will serve to confirm $^{113\text{m}}\text{In}$. The 255 keV γ -ray (decay from $E = 647$ keV to $E = 392$ keV) will confirm the presence of the parent, ^{113}Sn . At various timepoints, spectra of the purified daughter fraction and the purified parent fraction will be taken with an HPGe detector. Net peak areas of the 392 keV γ -ray and 255 keV γ -ray will be used to plot the decay/growth curves and confirm the parent-daughter relationship between the two nuclides.

Manufacturers of commercial Nuclear Medicine generator columns always report the percent breakthrough of parent activity into the daughter elution fraction. Generator columns are designed to minimize the breakthrough to prevent unnecessary radiation dose to the patient from the long-lived parent. One goal of the laboratory today is to characterize your column with regard to this parameter.

MATERIALS AND APPARATUS

1. An anion exchange column.
2. Chromatography resin (AG1 X8).
3. 4 N HCl, 2 N HNO₃
4. 4 N HCl solution containing $^{113}\text{Sn}/^{113\text{m}}\text{In}$ in equilibrium.
5. Three collection vials, a 1 mL pipette, parafilm.
6. 5 mL plastic counting vials.
7. Germanium gamma detector

PROCEDURES

CHEMISTRY

1. Column Preparation

- 1.1 Label 3 bottles: $^{113\text{m}}\text{In}$, ^{113}Sn , and H₂O wash.
- 1.2 Obtain resin and an empty column from the TA.
- 1.3 Place ~ 5 mL resin in a beaker containing ~ 10 mL 4 N HCl. Swirl to wet the resin.
- 1.4 Mount your column to the ring stand at a height such that the collection bottles may be placed beneath the column outlet. Pipette resin to the column and let resin settle to ~ the 3 mL mark. Place an empty beaker labeled "cold HCl waste" beneath the column.

- 1.5 Place a funnel on the column. Add 30 mL 4 N HCl and elute. Add additional resin if required to maintain 3 mL resin bed. Place a small amount of glass wool on top of the resin. The glass wool prevents the resin from being disturbed by the addition of further eluants. Elute until the liquid line just touches the top of the glass wool.
- 1.6 Cover the cold HCl beaker with parafilm.
2. **Elution of ^{113m}In**
 - 2.1 Pipette a volume containing 20 μCi of ^{113}Sn to the top of the column resin bed. Elute until the liquid level reaches the top of the glass wool.
 - 2.2 Transfer ~ 250 μL 4 N HCl to the column and elute until the liquid line reaches the top of the glass wool. **Do not let the column run dry!** Transfer an additional 15 mL of 4 N HCl to the column and elute. Collect all elutions in the bottle labeled " ^{113m}In ". **This is your purified daughter fraction.** Record the time when the elution is finished, this is your t_0 for your daughter fraction. Cap the bottle and set the bottle aside.
 - 2.3 Place the "H₂O wash" bottle beneath the column. Elute column with 6 mL of H₂O. Cap the bottle and set aside.
3. **Elution of ^{113}Sn**
 - 3.1 Place the " ^{113}Sn " bottle beneath the column. Elute column with 10 mL of 2 N HNO₃ to collect ^{113}Sn . **This is your purified parent fraction.** Record the time when the elution is finished, this is your t_0 for your parent fraction. Cap the bottle, mix well, wipe it with a tissue to ensure it is clean before proceeding.
 - 3.2 Pipette 2 mL of the purified parent (^{113}Sn) fraction into a pre-labeled 5 mL glass counting vial. One team member should have FST survey the counting vial immediately. **To observe the ^{113m}In growth curve, it is very important to start counting this fraction as soon as possible after the fraction has been collected.** That same team member should proceed with the parent isotope sample to the Counting Room to begin counting while other members continue with section below.
 - 3.3 Cap elution bottle and set aside.
4. **Further Sampling of the Column**
 - 4.1 Pipette 2 mL of the purified daughter (^{113m}In) fraction into a pre-labeled 5 mL counting vial. The second team member should have FST survey the

counting vial then proceed to the counting room with this sample for counting.
4.2 Cap elution bottles and set aside for cleanup.

5. **Clean-up**

While two team members are counting samples, the third will need to clean up the work area in the Chemistry Lab as per TA instructions. Radioactive bottles will be placed in the radioactive material storage hood. Acid waste will be placed in the chemical hood.

COUNTING SAMPLES AND DATA ANALYSIS

The Ge detectors will have certain shelves mounted that will accommodate your glass vials. If you must use a different position for your shelf mount due to dead time concerns or lack of activity, consult with the TAs. You will be using the Energy vs. channel calibration curve from your previous lab to identify which channel is due to the 392 γ ray peak and the 255 γ ray.

Measuring Secular Equilibrium Decay Kinetics

1. Count the purified parent fraction sample using the HpGe gamma detector as soon as possible after t_0 . The count time should be sufficient to collect > 1000 counts in the **392 γ ray peak. However, you will need to monitor both the 392 γ ray and 255 γ ray peaks, simultaneously.** The 392 γ ray peak will reflect the total activity (A_T), and you should expect to see values increase over time as the daughter ^{113m}In grows back in. **Record the relevant data from this peak in Table 1a.** The 255 γ ray peak will reflect the parent activity (A_P). **Record the relevant data in Table 1b.** The daughter activity from growth is calculated from the difference in A_T and A_P . **Record relevant data from these calculations in Table 1c.** Take additional data points (ie. spectra) at various time points spaced out across a 4 hour period. You will need roughly 8 data points to make a decent graph. The best method to accomplish this in GammaVision® is to set an ROI around the 392 keV peak and set the count length to stop when the area of the integral is > 1000 counts. **Try to keep the count time below 5 min or else you will run out of time and this will avoid applying additional timing corrections as described.** If the ^{113m}In decays by more than 10% during the counting time, i.e. for ^{113m}In , count times greater than 15 minutes, the net peak area should be corrected so that the gamma-ray emission rate is corrected to the start time of the count. The following equation gives the

multiplicative correction:

$$C_d = \frac{\lambda T_r}{1 - e^{-\lambda T_r}} \quad (9)$$

Where C_d is the correction factor for decay during the count time and T_r is the real (elapsed) time of the count.

2. On a single graph plot $\ln(A_T)$, $\ln(A_P)$ and $\ln(A_D)$ versus Elapsed Time (sec). Include error bars on all your data points. Using trend analysis in Excel and the best fit equation, project the growth of the daughter, $\ln(A_D)$, to equilibrium. What is your experimental equilibrium time.

Measuring Simple Decay Kinetics of ^{113m}In

1. Count the 392 keV peak in the “milked” ^{113m}In fraction at 30 minute intervals spanning over the same time period as above (roughly 8 time points). **Record relevant data in Table 2.** Each count time should be sufficient to collect > 1000 counts in the 392 keV peak. Note that spectra taken at later time points after the ^{113m}In daughter has sufficiently decayed should be analyzed for ^{113}Sn breakthrough at $E_\gamma = 255 \text{ KeV}$. Later you will be asked to assess how well your generator column performed. If you do not see a peak at 255 KeV it is below sensitivity limits of the detector.
2. Plot $\ln(A)$ from Table 2 versus elapsed time using Excel. Fit the data using linear regression analysis to calculate the half-life (from the slope) along with uncertainties. Report your equation and R^2 fit on the graph. Recall that the simple decay kinetics follows the equation:

$$A = A_0 \times e^{-\lambda t} \quad \text{therefore } \ln(A) = \ln(A_0) - \lambda t \quad (11)$$

Report the initial activity, A_0 , of the daughter (and the parent) at time t_0 . This is determined directly from the intercept of the equation.

3. ^{113}Sn Breakthrough Determination: Calculate a percent breakthrough of ^{113}Sn into the ^{113m}In fraction. If Gammavision® detected no activity in the 255 keV γ peak in your ^{113}In spectra, it will print an **MDA** value. MDA stands for Minimum Detectable Activity. The MDA value is a measure of how small an activity could be present and not be detected by the analysis. Think of it as an upper limit and use this value to calculate an upper limit on your percent breakthrough.

Additional Notes and Reminders:

1. Calculating Count Rate

The half-life of $^{113\text{m}}\text{In}$ is 99.5 min and the time of growth to equilibrium from the parent decay is relatively short. Therefore, the operations should be carried out quickly, but without rushing. The MCA software will record the count time from which the elapsed time intervals may be determined and the Net Peak Area. Remember that the counting time is the Live Time. **The count per second (cps) count rate is calculated as Net Peak Area \div Live Time.** Be sure to store your samples behind the shielding in the counting room when you are not counting so as to minimize background. Make sure that other people have also stored their samples away from the detectors.

2. Error Propagation

Errors must be propagated to report uncertainties using the following:

$$\sigma_R = \frac{\sqrt{x}}{t} = \frac{\sqrt{R \cdot t}}{t} = \sqrt{\frac{R}{t}} \quad \text{as such} \quad \sigma_{\ln(R)} = \ln\left(\frac{\sqrt{x}}{t}\right) = \ln\left(\sqrt{\frac{R}{t}}\right)$$

where σ_R is the standard deviation of the count rate (R) determined over the certain time (t) and $\sigma_{\ln(R)}$ is the standard deviation of the natural logarithm of the count rate.

POST-LAB DELIVERABLES

Notebook

- Include a brief statement of the lab's objectives.
- Include a sketch of your ion exchange setup including labels.
- Include of brief description of the procedures including counting of samples.
- Include copies of all your gamma spectra including calibration spectra and label all relevant parts.
- Include copies of Table 1 and Table 2.
- Include any observations from the lab that may have impacted your data.

Final Report

- Include a 1-page summary of what this lab was about (key elements we are looking for include title, date, lab partners, statement of objectives a brief statement on how these objectives were met through a description of the basic principles of ion exchange chromatography and how that was applied to demonstrate principles of secular equilibrium in Parent-Daughter decay.
- Include completed copies of Tables 1 & 2.
- Include plots of the following:

1. A copy of your HPGe energy calibration curve that you relied on for identifying critical peaks in today's lab.
 2. The natural log plot of your purified ^{113m}In activity with time fitted to a straight line using Linear Regression Analysis in Excel. Show labels and equation and calculate the half-life. Show error bars
 3. The natural log plot of your ^{113}Sn sample with time showing total activity, parent activity and daughter activity. Fit the daughter growth curve to a function and calculate the maximum time to reach equilibrium. Show error bars.
- Include a discussion of your results addressing the following:
 1. Discuss your column's performance based on expectations and include a discussion of your column's breakthrough (if any) of ^{113}Sn into the purified daughter fraction. Discuss what parameters in this lab might influence column performance.
 2. Discuss your secular equilibrium activity curves compared to theoretical expectations and discuss possible sources of error if your curves do not follow the activity-time relationships discussed in the Introduction.
 3. From your secular equilibrium plot, project how much time it should take for the ^{113m}In to grow back to its maximum activity? Calculate t_{max} using the following equation:

$$t_{\text{max}} = \frac{\ln(\lambda_2/\lambda_1)}{\lambda_2 - \lambda_1} \quad (12)$$

Discuss whether your experimental results match the theoretical time limit.
 4. In your discussion, include a statement of how long after ^{113m}In had been eluted from a generator would the column have to rest before it could be eluted again (assume you want maximum yield).
 5. Discuss your simple decay kinetics activity curve. How does your measured half-life compare with the actual value?
 6. Compare the projected daughter growth activity at equilibrium to the initial activity (A_0) of the purified daughter. Are they the same? Discuss reasons for any observed differences and changes you might effect in the experiment that might improve your data.
 - Include a summary statement of the merits of isotopes generators.

Table 1a. Raw Total Activity Data (A_T) from 392 KeV Peak from ^{113}Sn Sample.					
Elapsed Time (sec)	Net Peak Area	Live Time (sec)	Activity (cps)	$\ln(A_T)$	$\sigma \ln(A_T)$

Table 1b. Raw Parent Activity Data (A_P) from 255 KeV Peak from ^{113}Sn Sample.					
Elapsed Time (sec)	Net Peak Area	Live Time (sec)	Activity (cps)	$\ln(A_P)$	$\sigma \ln(A_P)$

Table 1c. Daughter Growth Activity (A_D) Calculated From Difference Between A_T and A_P				
Elapsed Time (sec)	$\ln(A_T)$	$\ln(A_P)$	$\ln(A_D)$	$\sigma \ln(A_D)$

Table 2. Raw Decaying Activity Data from 392 KeV Peak from “Milked” ^{113m}In Sample.					
Elapsed Time (sec)	Net Peak Area	Live Time (sec)	Activity (cps)	ln (A)	σ ln(A)

REFERENCES

1. G. B. Saha, Fundamentals of Nuclear Pharmacy, 4th ed., Springer (1998).
This book has a good discussion of generators and radioactive equilibrium.
2. J. Minczewski, J. Chwastowska, and R. Dybczynski, Separation and Preconcentration Methods in Inorganic Trace Analysis, John Wiley (1982).
3. K. A. Kraus and F. Nelson, Symposium on Ion Exchange and Chromatography in Analytical Chemistry, ASTM Special Technical Publication No. 195, Philadelphia, 1958, p. 27.
4. Taken from Laboratory Experiment No. 6 "Ion Exchange Chromatography" offered during CHEM 222, Quantitative Analysis Laboratory, Analytical and Environmental Chemistry Group at Washington State University.

Figure 1. A schematic of the chromatographic process. Note that the species represented by the triangle shows the least interaction with the stationary phase.

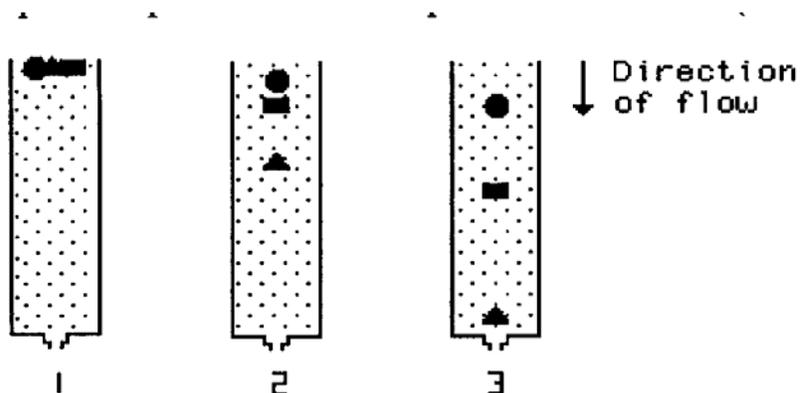


Figure 2. Anion-exchange separation of transition metals by stepwise elution development with HCl[1].

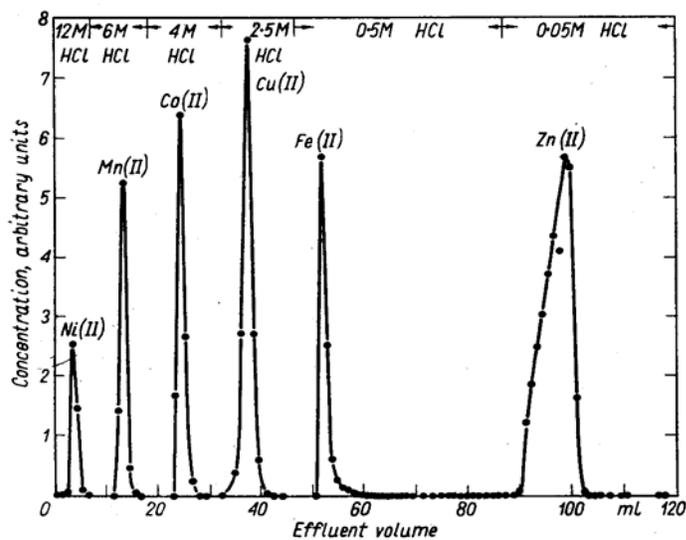




Figure 3. Styrene (A) and divinyl benzene (B) are copolymerized to form the support structure of many ion exchange resins.

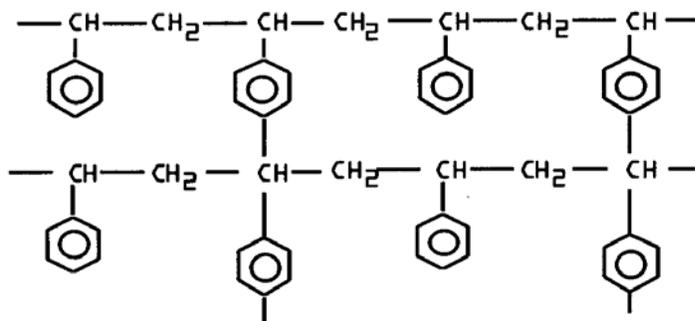


Figure 4. Representation of the backbone structure of ion exchange resins.

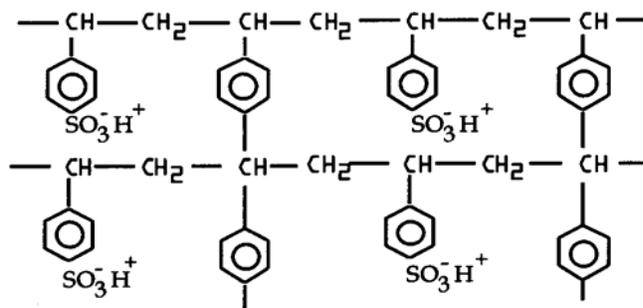


Figure 5. Sulphonic-acid cation-exchanger with a styrene-divinyl benzene matrix containing H^+ counter ions.

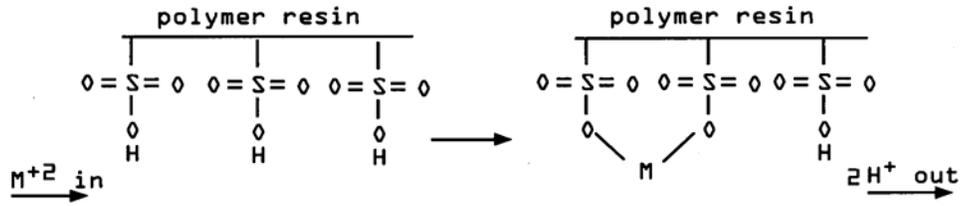


Figure 6. Representation of a divalent metal cation displacing H^+ counter ion on a sulphonic-acid cation-exchanger resin.

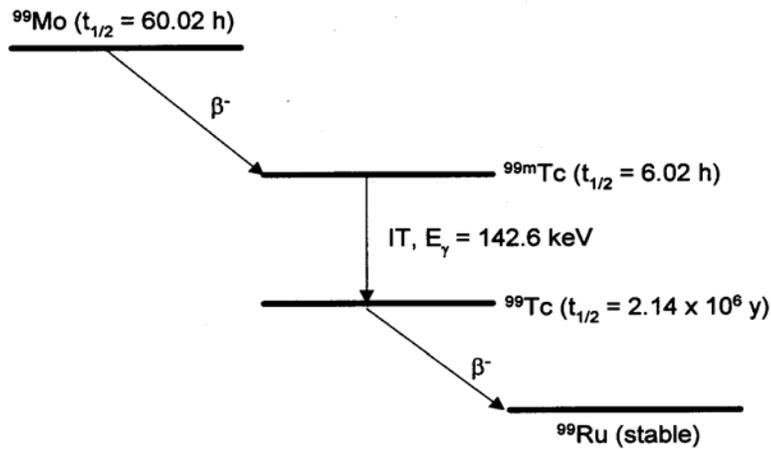


Figure 7. The $^{99}\text{Mo}/^{99m}\text{Tc}$ decay scheme used in the ^{99m}Tc generator (developed at BNL) and used in over 90% of all nuclear medicine procedures.

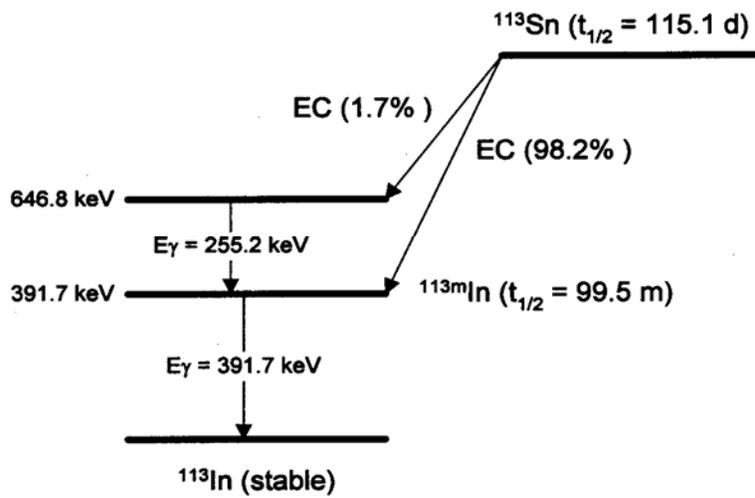


Figure 8. The $^{113}\text{Sn}/^{113}\text{In}$ decay scheme.

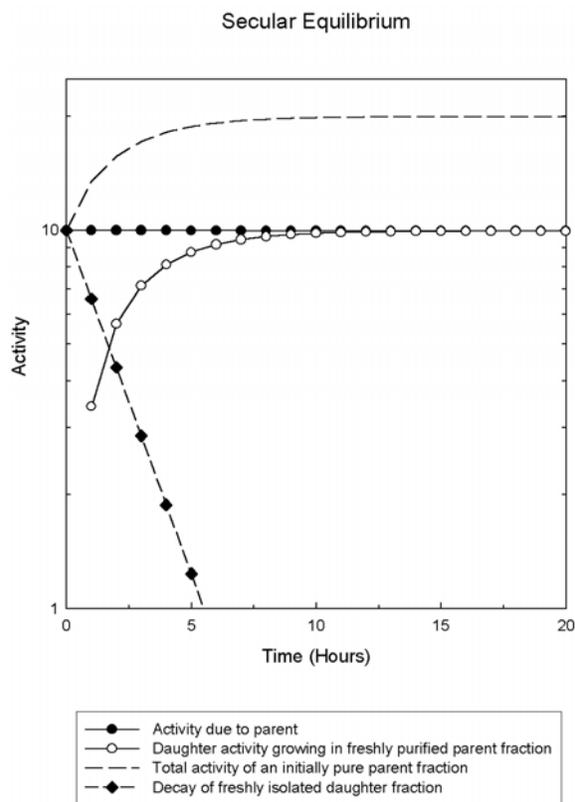


Figure 9. Deconvolution of an activity curve exhibiting secular equilibrium.

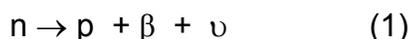
Liquid Scintillation Counting

OBJECTIVES

In Part 1 of today's lab, the objective will be to examine how certain types of chemicals present in your radioactive samples can influence the counting efficiency through either chemical or color quenching. In Part 2 of this experiment you will learn how the technique of spectral analysis works to compensate for sample quenching. In Part 3 you will apply spectral analysis to determine the absolute amount of radioactivity an unknown sample.

INTRODUCTION

Characteristics of β decay: β particles emerge from the nuclei of unstable atoms according to the reaction



where:

n = neutron in the nucleus

p = proton

β = beta particle whose mass and charge are exactly the same as an electron

ν = anti-neutrino

The energetics of this reaction, in general terms, are represented as follows:

$$E_n = E_p + E(\beta + \nu) \quad (2)$$

$$E(\beta + \nu) = E_\beta + E_\nu \quad (3)$$

All three terms of (2) are invariant with respect to a given isotope; hence, any values of E_β and E_ν are acceptable as long as their sum, $E(\beta + \nu)$ is invariant. Therefore, E_β may vary from $E(\beta + \nu)$ to 0 for values of E_ν ranging from 0 to $E(\beta + \nu)$ giving a distribution of E_β versus population.

Basic principles of measuring β decay by scintillation counting: A liquid scintillation counter does not count the β particles directly, but the photons of light emitted as the result of interaction of these β particles with the solvent and scintillator (fluor: compounds that give rise to fluorescence).

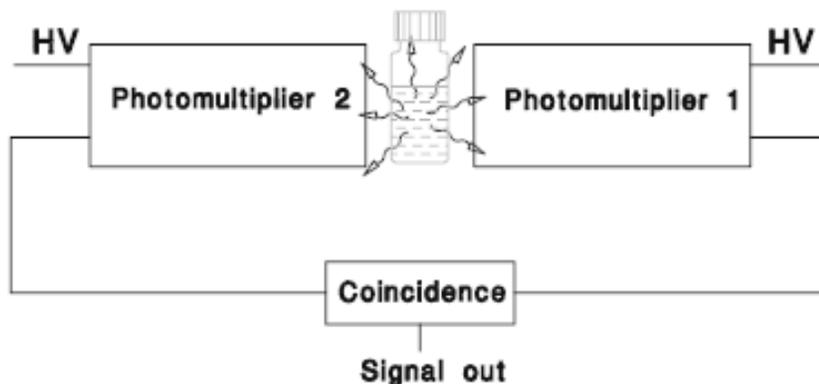
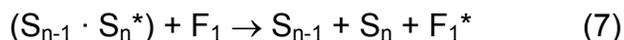
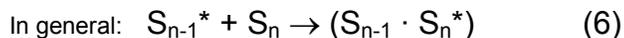
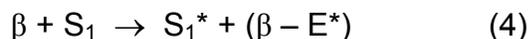


Figure 1. Block diagram of a liquid scintillation counter.

Because one measures light emission from the radioactive sample, stray light from outside the sample field-of-view (FOV) can often result in enormous background counts. To eliminate this effect most liquid scintillation counters are designed to operate using a coincidence circuit which only registers light detected simultaneously by two opposing photomultiplier tubes. This insures that any stray light from outside the sample FOV will not be registered as a light pulse.

The mechanism of energy transfer from a β particle to the fluor molecules (compounds that can give rise to fluorescence) in the mixed scintillator solution is complicated. Most of the β energy is absorbed in the solvent but a small fraction, through a series of steps, leads to electronic excitation of solute fluor molecules which de-excite by emitting photons. The following reaction diagram delineates the energy transfer scheme:



where:

S = solvent

S* = solvent in excited state

(S_{n-1} · S_n^{*}) = solvent eximer

F₁ = primary fluor

F₂ = secondary fluor

hν₁ = light output (3500-3800 Å)

hν₂ = light output (3900-4300 Å)

E* = energy lost by β particle during initial interaction

Note that steps 4-7 are radiationless, while steps 8-10 involve electromagnetic radiation. The number of photons emitted in this process is proportional to the β particle energy.

The liquid scintillation mixture, often called the “cocktail”, consists of a solvent (typically an aromatic solvent), a fluor (e.g. p-terphenyl), and the radioactive sample. Another compound (such as p-dioxane) is often added to the mix to increase the solubility of aqueous samples. Since many of the formerly used components are now considered toxic, new safer commercially available scintillation solutions are often used. The number of photons produced, and therefore the pulse size, is very sensitive to the exact composition of this “cocktail”.

Some chemical species introduced (intentionally or not) within the sample can often interfere with the energy transfer processes shown above and thus reduce the light output of the system. Quenching is a term used to describe the overall process by which the number of photons per β particle emitted is reduced. Quenching will manifest as an apparent shift in the β energy spectrum to lower energy (Fig 2).

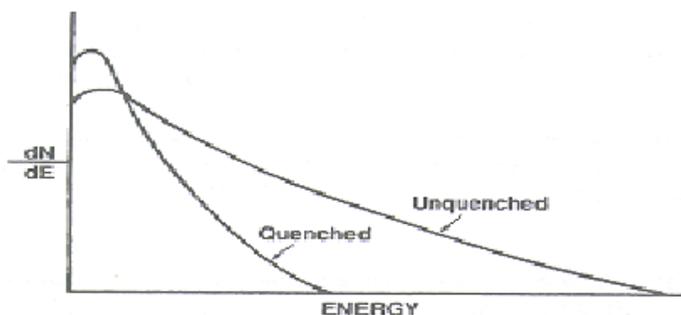


Figure 2. Energy spectrum of a decaying radionuclide.

Furthermore, quenching can occur on several fronts along the energy transfer pathway (see Fig 3).

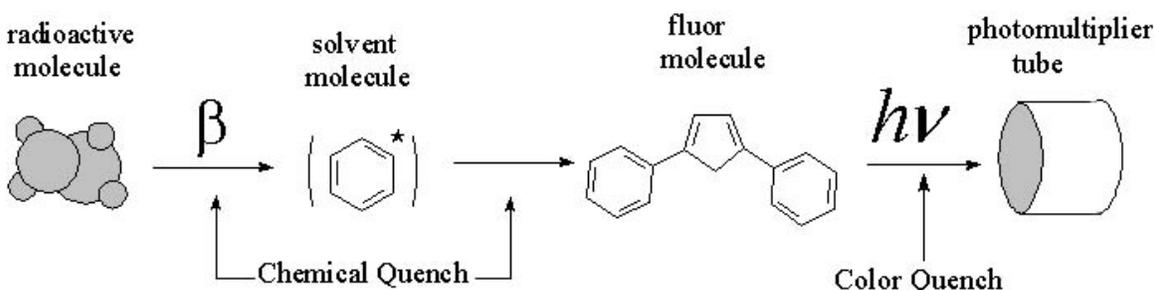


Figure 3. Quenching along the energy transfer process for scintillation counting.

Chemical quenching: This form of quenching influences the energy transfer pathway **before** the production of photons. It most often occurs during the transfer of energy from the solvent to the scintillator, but can also uncouple excitation of the solvent. Any chemical species that is highly electronegative (ie. electron capturing) will effect the energy transfer process by capturing or stealing the π electrons from the solvent molecule that are necessary for efficient energy transfer. Other mechanisms for chemical quenching can involve chemical alteration of the sample components. For example, when an alkaline sample is added to the p-dioxane scintillation cocktail mixture, the dioxane can become oxidized to an epoxide, thus eliminating the solvent from participation in the energy transfer process. In other cases, the solvent eximer can transfer it's excitation energy to a material in the sample which, once excited, relaxes to it's ground-state radiationlessly, thus converting the initial energy of β decay to heat and motion.

Color quenching: This form of quenching influences the energy transfer pathway **after** the production of photons. It occurs by absorption of light by a molecule (quenching agent) when that molecule's resonance transition equals that of the incident photon energies listed in steps 8 & 10. At the macroscopic level, this process is becomes concentration dependent. Otherwise stated, as the concentration of the quenching agent increases, the number of photons detected by the system decreases. Since the fluorescent materials used in liquid scintillation counting emit photons whose energies range from 365-480 nanometers, other materials will behave as quenching agents if they absorb light in this same wavelength range.

Dilution quenching: A third less important form of quenching is call dilution quenching. Anything which separates the "transferors" of energy in the system is considered a dilution quencher; therefore, all the components of the sample may be considered to be dilution quenchers. Care must be taken in nuclear assay that all samples (including standards) are diluted to nearly equivalent volumes.

Compensating for Quenching: Since it is oftentimes desirable to be quantitative when performing a nuclear assay, a means for determining the relationship between quench and counting efficiency must be determined. In the second part of today's lab you will explore a technique known as **Spectral Analysis** that enables quantitative correction for effects of quenching on the counting efficiency.

Most modern integrated liquid scintillation systems are equipped with the ability to perform quench corrections using spectral analysis. However, there are several variations on this general approach depending on what is measured. One variation relies on the measured **Spectral Index of the Sample (SIS)**. The SIS value decreases as sample quenching increases, reflecting the shift of the spectrum to lower energy (Fig. 1). A second variation relies on measuring the transformed **Spectral Index of the External Standard (t-SIE)**. This index is calculated from the Compton spectrum induced in the scintillation cocktail by an external ^{133}Ba gamma source (most modern instruments come equipped with an internal gamma source for this purpose). The source is positioned under the sample vial, causing a Compton spectrum to be produced within the cocktail solution. From a mathematical transformation of this spectrum, the t-SIE value can be determined. Like SIS, t-SIE values decrease as sample quenching increases. Of the two spectral approaches, the t-SIE method is typically more useful for several reasons: (1) it is independent on the nature of the sample isotope; (2) it is independent of the activity level in the vial; and (3) it has a broad dynamic range. SIS uses the sample isotope spectrum to track quenching; it is only accurate for high-count rate samples. Additionally, the range of SIS values reflects the energy of the sample isotope. Hence, its dynamic range is not as great as t-SIE.

In Part 2 of today's lab you will demonstrate some of the basic principals of SIS by following the change in the ratio of sample counts falling in two counting channels on the liquid scintillation instrument and the corresponding change in the efficiency. Figure 4 shows the count distribution in two channels, labelled A and B. Several examples of a quench count distribution are also shown. It is clear that the ratio of counts in channel B to those in channel A will decrease as quenching increases (ie. Counting Efficiency decreases). Figure 5 is a graphical combination of these facts. Such a curve is generated from data gleaned from a set of standards, where each member of the set has a different amount of quenching agent and a known amount of radioactivity. When samples of unknown dpm are counted and an attempt to correct for quenching is made by this method, the windows used to set up the calibration curve must also be used for the sample count. For today's lab these parameters will be set for you by the TA's.

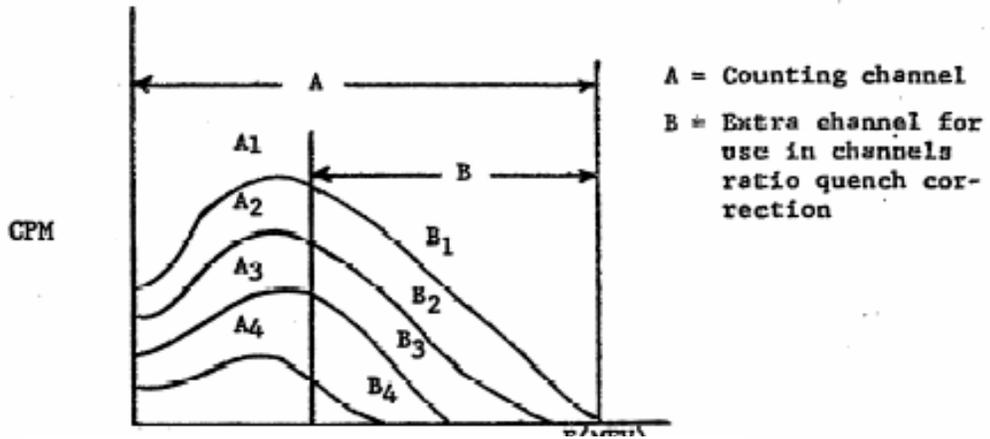


Figure 4. Shows again the energy shift in the sample β spectrum at 4 levels of quenching. Note how activity within designated channels A and B change.

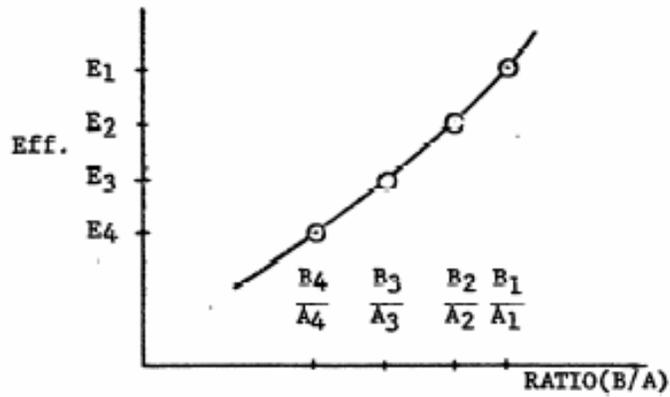


Figure 5. Correlation of counting efficiency with B/A ratio.

APPARATUS AND MATERIALS

1. Liquid Scintillation Analyzer (Tri-Carb 1600)
2. Several microliters of activity bearing solution:
 - a. Teams 1 & 2 will use [^3H]acetate
 - b. Teams 3 & 4 will use [^{14}C]acetate
3. 22 scintillation vials
4. ~110 mL scintillation cocktail in autodispenser (Ultra-Gold)
5. 2 mL of appropriate chemical quenching agent for Part 1.
 - a. Teams 1 & 3 will use acetone
 - b. Teams 2 & 4 will use nitromethane
6. 2 mL of appropriate color agent for Part 1. (prepared by TA's)
 - a. Teams 1 & 3 will use methyl orange solution (0.00001%)
 - b. Teams 2 & 4 will use methylene blue solution (0.00001%)
7. 2 mL of nitroethane quenching reagent (all teams) for Part 2.
9. Unknown radioactive sample (either ^3H or ^{14}C depending on Team)

Part 1. PROCEDURES

Note that the final write up of this lab will involve coordination of data between all 4 teams. Teams 1 & 2 are assigned to work only with [^3H]-acetate as their radioactive source. Teams 3 & 4 are assigned to work only with [^{14}C]-acetate as their radioactive source. The final write up will require interpretation of all data to determine how the different quenching agents affect the counting efficiencies of different beta emitting radioisotopes. After the lab the class will meet to exchange data and discuss trends before final write-up.

Prepare the following array of 15 samples by first dispensing 5 mL of scintillation cocktail mix into the vials. For the first part, you will establish what your standard radioactivity is (in triplicate and calculating the mean value) by pipetting a constant amount of radioactivity within each standard sample, ie ~30,000 dpm of ^3H or ~60,000 dpm of ^{14}C depending on your team assignment. Each team will be given a vial of radioactivity (~2 mL total volume) from which to draw aliquots using their 1000 μL pipettor. The dose will be adjusted so that 100 μL will yield close to the desired count rate within each vial. **Use a 100 μL volume of radioactivity for all of your samples.** In the second part of this study, each team will prepare a series of samples containing the appropriate chemical and color quencher assigned to them. Each team will use the sample preparation protocol outlined in Table 1. Note that the quenching agent should be introduced into the vial before the radioactivity using the 100 μL pipettor and changing tips per sample (ie. the order of introduction is: scintillation cocktail>quenching agent>radioactivity)

Once all the reagents are introduced, tightly cap the vials and label them. To avoid confusion when samples are loaded into the counting racks that the tops of the caps be affixed with the appropriate colored label with the sample

identifier as indicated in the shaded portion of Table 1. Also, to ensure uniform mixing of radioactive sample, quenching agent and scintillation cocktail mix, gently swirl each vial before counting.

Table 1.					
Team 1: ³H-radioactivity					
Sample #	Standard	Sample #	Methyl Orange (μL)	Sample #	Acetone (μL)
1A1	---	1B1	10	1C1	5
2A1	---	2B1	20	2C1	10
3A1	---	3B1	30	3C1	15
		4B1	40	4C1	20
		5B1	50	5C1	25
		6B1	60	6C1	30
Team 2: ³H-radioactivity					
Sample #	Standard	Sample #	Methylene Blue (μL)	Sample #	Nitromethane (μL)
1A2	---	1B2	10	1C2	5
2A2	---	2B2	20	2C2	10
3A2	---	3B2	30	3C2	15
		4B2	40	4C2	20
		5B2	50	5C2	25
		6B2	60	6C2	30
Team 3: ¹⁴C-radioactivity					
Sample #	Standard	Sample #	Methyl Orange (μL)	Sample #	Acetone (μL)
1A3	---	1B3	10	1C3	5
2A3	---	2B3	20	2C3	10
3A3	---	3B3	30	3C3	15
		4B3	40	4C3	20
		5B3	50	5C3	25
		6B3	60	6C3	30
Team 4: ¹⁴C-radioactivity					
Sample #	Standard	Sample #	Methylene Blue (μL)	Sample #	Nitromethane (μL)
1A4	---	1B4	10	1C4	5
2A4	---	2B4	20	2C4	10
3A4	---	3B4	30	3C4	15
		4B4	40	4C4	20
		5B4	50	5C4	25
		6B4	60	6C4	30

Count all samples in a wide open window for 1 minute each (Channel A on printout), and record data using Table 2. Note that each team will have a variation of Table 2—teams will need to exchange their Table 2 data with other teams in order to have a complete data set for final write-up.

For the purpose of filling in the last column of this data table you must use the mean (and standard deviation) of the three replicates you counted of the standard. Note that you can assume dilution effects will be minimal so the mean standard value will be used to calculate the efficiency for all the additive samples. **At the completion of this lab each individual should have 4 versions of Table 2 data reflecting a compilation of all team data.**

Part 2 & 3. PROCEDURES

Spectral Analysis Method: For this part of the lab you will be examining shifts in the β energy spectrum as measured across two channels in the liquid scintillation counter. You will need to prepare 5 scintillation vials containing 5 mL of scintillation fluid, varying amounts of **nitroethane** and a fixed (100 μ L) volume of your radioactive source, which is the same as in Part 1. **You will rely on the mean standard cpm value obtained in Part 1 and will consider that value to be the absolute dpm—this assumes the intrinsic counting efficiency of the instrument is 100% for both ^3H and ^{14}C .** Use Table 3 (column 1) as a guide for labeling vials with your colored dots.

Count the samples and record data in Table 3. using two window settings on the liquid scintillation counter (see Figure 2). **Note: channel A is wide open; channel B is 70% of channel A. The 70% window will be set by the instructor where the upper discriminator will be maintained at a constant level and the lower discriminator will be increased to obtain the appropriate discrimination.**

Obtain an unknown sample from the instructor, which will have a certain varying amount of nitroethane quencher and 100 μ L of the radioactive standard solution and 5 mL of scintillation cocktail. Count the unknown sample using the **same** two windows in which the samples were counted, and fill in Table 4. The Efficiency in column 5 is determined from plot of Efficiency vs. B/A using data from Table 3 to construct this plot (see Data Analysis Section).

DATA ANALYSIS

Part 1:

1. Using the data from the compilation of Table 2 (all team data), construct four (4) plots of the calculated % Efficiency (Y-axis) vs. Volume of Quenching Agent (X-axis). Each plot should have the respective data for the chemical or color quenching agent and either ^3H or ^{14}C . Simply put, a sample plot will have two curves showing the effects of nitromethane and acetone on chemical quenching of the ^3H radioactive sample. Include error bars as uncertainties in your calculated % Efficiency. Also, each plot should be accompanied by a legend indicating what data is associated with what reagent.

Part 2:

1. Using data from Table 3 construct a single plot of the B/A Ratio vs. Volume of Quenching Agent. Include error bars.
2. Using data from Table 3 construct a single plot of % Efficiency vs. B/A Ratio. Include error bars (both as uncertainties in your calculated % Efficiency and B/A ratio—that is include Y- and X-error bars).
3. Calculate the absolute activity in your unknown sample using the following formula:

$$\text{Absolute Activity (dpm } \pm \sigma) = \text{cpm } \pm \sigma \text{ (in channel A)} \div \text{fractional Efficiency}$$

POST-LAB DELIVERABLES

Notebook

- Include a brief statement of the lab's objectives.
- Include a block diagram of the components of a liquid scintillation counter.
- Include of brief description of the procedures including counting of samples.
- Include copies of Tables 2-4.
- Include any observations from the lab that may have impacted your data.

Final Report

- Include a 1-page summary of what this lab was about (key elements we are looking for include title, date, lab partners, statement of objectives a brief statement on how these objectives were met through a description of the basic principles of liquid scintillation counting and how sample integrity can influence the "quality" of the data you measure.
- Include completed copies of Tables 2-4.

- Include plots of the following:
 - a. Four (4) plots of the calculated % Efficiency (Y-axis) vs. Volume of Quenching Agent (X-axis). Each plot should have 2 curves on it.
 - b. Plot of the B/A Ratio vs. Volume of Quenching Agent.
 - c. Plot of % Efficiency vs. B/A Ratio.
- Include a discussion of your results addressing the following:
 - a. Discuss any differences you observe between color and chemical quenching agents. Is any one mode of quenching stronger than the other?
 - b. Within individual “classes” of quenching agents (ie. color and chemical) what conclusions can you draw on relative strengths of these agents. Hint (1): for color quenchers consider how differences in each molecule’s absorbance wavelength and extinction coefficient reflect any trends you observed in quenching (see the Table below for guidance). Hint (2): for chemical quenchers consider how differences in their electronegativities reflect any trends you observe in quenching.

Dye	Maximum Absorbance Wavelength (nm)	Extinction Coefficient at Maximum Absorbance Wavelength ($\times 10^3$)
Methylene blue	656	82
Methyl orange	460	27

- c. Referring to your plots of % Efficiency vs. Vol. of Quenching Agent, discuss how sensitivity to quenching agent might be dependent on the nature of the isotope being measured (ie. E_{β})
- Include a brief summary statement that takes in to account the results on your unknown sample (ie. how close was your result to the actual value). Determine the volume of nitroethane in your unknown sample from your plotted data of B/A vs. volume nitroethane (how close was your result to the actual volume that it should be—TA will provide the actual amount). Discuss the merits/pitfalls of Spectral Analysis as a correction method. Consider the accuracy of the approach for samples quenched to a very low count rate.

REFERENCES

Hawkins EF “Scintillation Supplies and Sample Preparation” Donald L. Horrocks Nuclear Applications Laboratory Beckman Instruments, Inc.

Table 2.
Data Set: Team []

Sample #	Sample composition	CPM	Mean	Stdev
1A[]	---			
2A[]	---			
3A[]	---			
Sample #	Sample composition	CPM	(CPM Sample÷CPM Standard) x 100 % Efficiency	σ
1B[]				
2B[]				
3B[]				
4B[]				
5B[]				
6B[]				
1C[]				
2C[]				
3C[]				
4C[]				
5C[]				
6C[]				

Table 3.

Standard #	cpm in A (wide-open window)	σ	% Efficiency 100 x (A/dpm)	Error	cpm in B (70% window)	σ	B/A Ratio	Error
1E[]								
2E[]								
3E[]								
4E[]								
5E[]								
6E[]								

Table 4.

Unknown Sample	cpm in A (wide open window)	σ	cpm in B (70% window)	σ	Calc. B/A Ratio	Error	% Eff.	Calc. Activity (dpm)	Error
1F[]									

Radiochemical Separation of $^{59}\text{Fe}(\text{III})$ by Solvent Extraction

Objectives

The objective of the experiment today is to demonstrate some simple principles underpinning solvent extraction. You will be calculating the extraction coefficient, K_D , for extracting iron(III) from an aqueous solution, and from that the parameter the percent extraction, %E. The percentage of iron extracted will be determined using the radioisotope ^{59}Fe ($t_{1/2}$, 45 day; β^- energies, 0.46 and 0.27 MeV; γ energies, 1.10 and 1.28 MeV). Iron(III) will be extracted from the aqueous solution into an organic solvent as an alkyl phosphate complex. You will test two parameters influencing K_D (and ultimately the extraction efficiency) including $[\text{H}^+]$ and $[\text{Fe}^{+3}]$.

Background

Liquid-liquid (or solvent) extraction is a technique that allows for selectively transferring a species M^{+n} between an aqueous solution and an organic phase that typically possesses a low dielectric constant and weak hydrogen bonding capacity (typically aromatic and chlorocarbon solvents have been used) by equilibrating the aqueous phase with an organic solvent. Usually the organic phase also contains an extractant which is capable of forming a neutral compound MA_N with the species M^{+n} to be transferred between phases. The number of demonstrated extractants used are numerous; typical extractants include organophosphates (such as di(2-ethylhexyl)phosphoric acid or tri-n-butyl phosphate), various ketones (such as methyl isobutyl ketone) and various amines (typically highly branched primary amines).

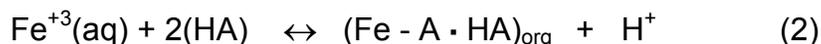
In order for the extraction process to work, a solute must be distributed between two immiscible liquids. Typically, these liquids are placed into a separatory funnel (see Fig 1) where they are shaken vigorously to allow adequate contact of the solute with all phases. However, the liquids will quickly part into two immiscible phases once the funnel is allowed to sit. Due to variation in chemical properties between the two liquids, one liquid will dissolve more solute than the other. A number of factors play a controlling role in solubility and this is largely dictated by the Gibb's free energy of the system described by Equation 1:

$$\Delta G = \Delta H - T\Delta S \quad (1)$$

The first term ΔG is the change in free energy, ΔH is the change in the enthalpy, T is the temperature, and ΔS is the change in entropy (randomness). In order for dissolution to occur, the change of free energy of the system must be negative (ref 1). This requires that the process (including any chemical modification to the solute) not be too highly endothermic in order to keep ΔH small, while a high ΔS is

favorable. A number of other factors contribute to the overall solubility of a system including lattice energy which dictates the separation of the solute into individual molecules (this is typically endothermic, but small when solute molecules are nonpolar), interactions between the solute and solvent molecules (this is typically exothermic, but again small if both solvent and solute are nonpolar). Hence, for a given solvent extraction to be successful, a solvent and aqueous solution must be selected that will take advantage of some of these principles.

Furthermore chemical modification of the solute can most often lead to enhanced and selective solubility in the organic phase. In today's experiment you will be performing extractions using toluene as the organic solvent and di(2-ethylhexyl) phosphoric acid (HA) as the extractant. The general scheme of complexation involves the following reaction:



Things that will affect this equilibrium include the concentration of Fe^{+3} , HA and H^+ . The later parameter is especially useful for fine tuning extraction selectivity when dealing with a mixture of elements. For example, Figure 1 demonstrates how one can effectively separate a number of divalent states of certain metals by changing the starting pH of the aqueous phase. That is, a mixture of Fe^{+2} and Mn^{+2} can be easily separated if the extraction is run in very acidic conditions.

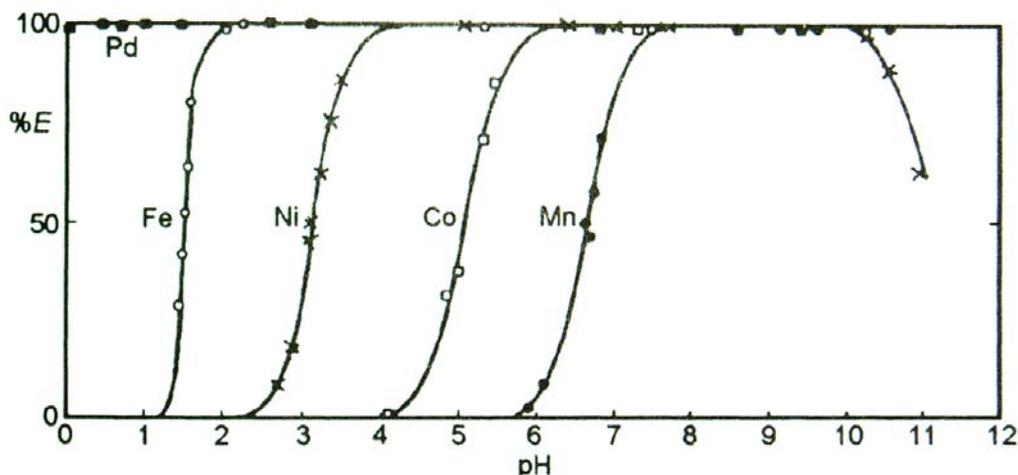


Figure 1. Effect of pH on the extraction of some divalent metals from aqueous solution into chloroform by 0.01M 8-hydroxyquinoline (oxime).

Some Terms to Consider: If the Distribution law defines the equilibrium reaction $\text{A}(\text{aq}) \leftrightarrow \text{A}(\text{org})$ then the Distribution Coefficient, K_D , can be defined as:

$$K_D = \frac{\text{Total metal concentration in the organic phase}}{\text{Total metal concentration in the aqueous phase}} \quad (3)$$

$$K_D = [M]_{\text{org}} / [M]_{\text{aq}} \quad (4)$$

The Recovery Factor, %E, is defined as the percentage of the total quantity of substance extracted under specific conditions. %E is related to the Distribution Coefficient by:

$$\%E = \frac{100K_D}{K_D + (V_{\text{aq}}/V_{\text{org}})} \quad (5)$$

where V_{aq} and V_{org} are the volumes of the aqueous and organic phases, respectively. When volumes are equal then Eq. 5 reduces to the following:

$$\%E = \frac{100K_D}{K_D + 1} \quad (6)$$

Figure 2 shows how %E relates to K_D . This figure shows that the %E method of showing extraction data is a more useful description of the success or failure of the extraction. For example, the wide range of $99 \leq K_D \leq \infty$ has %E in the narrow range of 99-100% and would be considered a successful extraction.

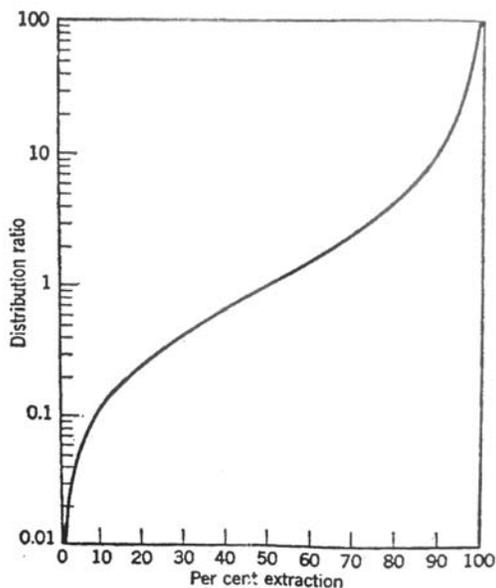


Figure 2. Relation of K_D to the %E for a particular solvent extraction system.

Apparatus and Materials

1. Tri Carb 1600 Liquid Scintillation Counter
2. Ultra-Gold Scintillation cocktail mix (~75 mL per team)
3. Scintillation counting vials (12 per team)
4. toluene (~100 mL) and acetone (~50 mL)
5. di(2-ethylhexyl)phosphoric acid (DEHPA) as 0.5 N solution in toluene
6. HCl (4 N)
7. FeCl₃
8. ⁵⁹Fe⁺³ radioactive solution (12 mL aqueous solution at ~ 1 μCi per mL)
9. graduated cylinders, beakers, 50 mL plastic bottles
10. 30 mL separatory funnel (with ring stand and support clamp)
11. pouring funnels
12. 250 mL plastic bottles: label one as ⁵⁹Fe-aqueous waste and the other as ⁵⁹Fe-organic waste.

Procedures

Note 1: You will be handling radioactive aqueous and organic solutions. Be very careful to place all equipment within the yellow tray in order to minimize the chance for contamination to occur outside your dispersibles work area. **Treat anything within this zone as being contaminated (wear proper PPE prior to handling any items in this zone—always wear nitrile or latex disposable gloves when handling solutions or equipment, and dispose of these gloves when you are finished with each step—donning fresh gloves as needed).**

Additional notes for controlling dispersible radioactive materials:

- Always remove liquids from the separatory funnel through the stopcock at the bottom. Never pour liquid out through the top as this could splash or dribble causing a contamination.
- After each extraction, wipe the outside of the funnel using Kim wipes checking at each stage of cleaning using a portable frisker. Dispose of any Kim wipes into your radioactive waste receptacle.
- Additional PPE requirements--we will require that you wear leave protectors (gauntlets) in addition to a lab coat, gloves and shoe covers (paper booties). Treat all these additional PPE items as disposable.

Note 2: TA's will provide you with the standard ⁵⁹Fe activity per mL volume.

Effect of [H⁺] on ⁵⁹Fe⁺³ Extraction:

1. Prepare the following acid solutions, ~10 mL each (0.1 N, 0.2 N, 0.5 N, 1 N, 2 N and 4 N HCl. Use graduated cylinders to measure acid and dilute with water. You will need to recalculate the change in your HCl normality once you have added the additional 1 mL of radioactive water to your 9 mL of acid solution—this is important in final graphics.
 - 1.1 Add water to the cylinder first then add the appropriate volume of acid.
 - 1.2 Stir the contents in the cylinder using a glass stirring rod.
 - 1.3 Pour contents of cylinder into a labeled plastic bottle.
2. Close the stopcock on the 30 mL separatory funnel. Add 9mL of the appropriate acid solution to the funnel. Tighten the stopcock at this point if you notice any leakage.
3. Add 1 mL of your ⁵⁹Fe stock solution using your 1000 μL pipetter. (Remember to dispose of the pipette tip in the radioactive waste receptacle.)
4. Add 10 mL of 0.5 N DEHPA solution in toluene using a graduated cylinder. Cap funnel and gently shake to mix contents for a few minutes.
5. Set the funnel back into its ring stand and carefully remove cap using a Kim wipe to cover over it. **Cap is now contaminated!** Have one of the team members clean the cap by rinsing it using water bottle—hold cap over a beaker during this process to catch the rinse. Dispose of this rinse in the radioactive ⁵⁹Fe-aqueous waste receptacle.
6. Place the radioactive waste receptacle underneath the separatory funnel outlet—open stopcock and collect the aqueous phase. (**Remember that the organic phase is on top.**) Be sure not to let the toluene phase dribble into this fraction.
7. Next collect the organic phase in a 50 mL beaker.
8. Pipette 0.5 mL of the organic phase into a scintillation vial and add 10 mL of scintillation cocktail mix to it. Cap vial and smear outside using a Kim wipe to ensure it is not contaminated. Swirl the capped vial to get uniform mixing. Label the outside of the vial before setting it up for counting.
9. Dispose of the remainder of the toluene phase in the radioactive waste bottle labeled ⁵⁹Fe-organic waste.
10. Close the stopcock and add 5 mL of pure toluene to the funnel, cap, and swirl to rinse remaining radioactivity from the inside surface. Collect the rinse through the stopcock and add this to the radioactive organic waste receptacle. (**Always cap the organic waste receptacle when you are not adding material to it—**

toluene is quite volatile and inhaling the fumes can be harmful to your health.)

11. Repeat this cleaning step using 5 mL of acetone. **Add this rinse to the radioactive aqueous waste receptacle.**
12. Check your gloves and gauntlets for contamination. Change if necessary.
13. Repeat steps 1-12 for the other acid solutions your team has been assigned.

Effect of $[\text{Fe}^{+3}]$ on $^{59}\text{Fe}^{+3}$ Extraction:

Note: all extractions for this part of the experiment will be performed using an aqueous phase of 1 N HCl.

1. Prepare the following FeCl_3 solutions by weighing out to following milligram amounts of salt using the analytical balance and dissolving them each in 10 mL of 1 N HCl (5 mg, 10 mg, 15 mg, 20 mg, 30 mg and 40 mg). You will need to calculate the molar concentration of Fe^{+3} for each solution. Don't forget to correct for dilution when you add 1 mL of radioactive ^{59}Fe and ignore any iron mass from adding the radioactive sample as it will be insignificant.
2. Close the stopcock on the 30 mL separatory funnel. Add 9 mL of the aqueous acidic solution to the funnel that contains the appropriate amount of iron carrier. Tighten the stopcock at this point if you notice any leakage.
3. Add 1 mL of ^{59}Fe stock solution (obtained from the TA) using your 1000 μL pipetter. (Remember to dispose of the pipette tip in the radioactive waste receptacle.)
4. Add 10 mL of 0.5 N DEHPA solution in toluene using a graduated cylinder. Cap funnel and **gently** shake to mix contents for a few minutes.
5. Set the funnel back into its ring stand and carefully remove cap using a Kim wipe to cover over it. **Cap is now contaminated!** Have one of the team members clean the cap by rinsing it using your water wash bottle—hold cap over a beaker during this process to catch the rinse. Dispose of this rinse in the radioactive ^{59}Fe -aqueous waste receptacle.
6. Place the radioactive waste receptacle underneath the separatory funnel outlet—open stopcock and collect the aqueous phase. (**Remember that the organic phase is on top.**) Be sure not to let the toluene phase dribble into this fraction.
7. Next collect the organic phase in a 50 mL beaker.
8. Pipette 0.5 mL of the organic phase into a scintillation vial and add 10 mL of scintillation cocktail mix to it. Cap vial and smear outside using a Kim wipe to

ensure it is not contaminated. Gently swirl to get a uniform mix. Label the outside of the vial before setting it up for counting.

9. Dispose of the remainder of the toluene phase in the radioactive waste bottle labeled ^{59}Fe -organic waste.
10. Close the stopcock and add 5 mL of toluene to the funnel, cap, and swirl to rinse remaining radioactivity from the inside surface. Collect the rinse through the stopcock and add this to the radioactive waste receptacle. **(Always cap the toluene waste receptacle when you are not adding material to it—toluene is quite volatile and inhaling the fumes can be harmful to your health.)**
11. Repeat this cleaning step using 5 mL of acetone. Add this rinse to the radioactive aqueous waste receptacle.
12. Check your gloves and gauntlets for contamination. Change if necessary.
13. Repeat steps 1-12 for the other aqueous solutions your team has been assigned.

Counting

You will use the Packard Tri Carb 1600 liquid scintillation counter to assess levels of radioactivity in your organic phase samples. The instrument will be set to a full open window and you will get your data from the Channel A printout. For efficient use of time all the samples should be staged for counting at the end of all the extractions. Your TA will assist you in this.

DATA TABLE

[HCl]	[Fe ³⁺]	[M] _{aq} = (R _T - R _{org})	[M] _{org} = R _{org}	K _D	%E
	--				
	--				
	--				
	--				
	--				
	--				
1 N					
1 N					
1 N					
1 N					
1 N					
1 N					

R_T is the total activity of ^{59}Fe in your extraction system based on your standard. Use the mean value from all 4 team's standards.

R_{org} is the amount of activity in your organic extract—don't forget to perform a fraction correction for the total volume of your organic phase.

$[\text{HCl}]$ can be expressed as normality of solution.

$[\text{Fe}^{+3}]$ can be expressed as molar concentration. In Part 1 (where you vary $[\text{H}^+]$) assume $[\text{Fe}^{+3}]$ is insignificantly small for the no carrier added parts of their studies.

Clean up

1. Place an absorbent diaper alongside your yellow tray. Cap all waste bottles and wipe the outside surfaces using Kim wipes until clean—check this with the frisker. Once clean place the bottles on the diaper.
2. Wipe clean the outside of the separatory funnel following the same procedures as above and place on the diaper. (Note that you have already rinsed out the inside of the separatory funnel.)
3. Consolidate any solid radioactive trash (pipette tips, Kim wipes etc.) into your solid waste receptacle. Clean outside of the receptacle using Kim wipes and place it back in your tray.
4. Wipe all surfaces in your work area.

Notebook

- Include a brief statement of the lab's objectives.
- Include a sketch of your experimental setup.
- Include a brief (1 paragraph) procedure.
- Include copies of the Table for your raw data.
- Show a record of mass measurements.
- Include any observations from the lab that may have impacted your results.

Lab Report

Use the standard report format and include the Table of your data and the following plots.

- %E vs. $[\text{HCl}]$ as normality and comment on any trends.
 - %E vs. $[\text{Fe}^{+3}]$ as molarity and comment on any trends.
 - Construct a plot of K_D vs. %E using all your data.
1. Based on your previous experiences using liquid scintillation counting discuss why it was necessary to count a standard for your ^{59}Fe that had the same amount of DEPH complexation reagent rather than measure $[\text{M}]_{\text{aq}}$ directly.

2. As a follow-up to 1, use your knowledge of other nuclear detection methods, and discuss one other way you could have quantified your extraction results.
3. Comment on the trends you observed from plots of %E vs. [HCl] and [Fe⁺³]. Do these trends fit with expected shifts in chemical equilibrium?
4. Comment on the general shape of your plot of K_D vs. %E. Does it look anything like Figure 2?
5. Discuss advantages and/or limitations of this approach for possible selective extraction of a mixture of metal ions in aqueous solution.

REFERENCES

A good on-line reference on solvent extraction can be found at:

<http://ull.chemistry.uakron.edu/chemsep/extraction>

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Nucleophilic Aromatic Substitution using [^{18}F]-Fluoride Ion

OBJECTIVES

Students will assess the effects of (1) temperature and (2) nature of leaving group on reaction rate by measuring the extent of [^{18}F]-fluoride ion labeling *via* nucleophilic aromatic substitution.

BACKGROUND

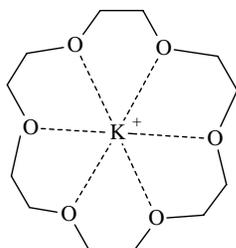
The element fluorine is in many ways unique, both in its chemical characteristics and usefulness in the pharmaceutical and chemical industries. Fluorine has a very small steric size and exhibits very high carbon-fluorine bond energies; fluorine is also extremely electronegative, and such substitution can often produce significant and useful changes in physiological, biological and chemical properties of organic compounds. For example, polyfluorinated organic compounds such as Teflon and Freons are noticeably non-reactive toward chemical reactions hence are used for creating inert chemical surfaces or environments.

Fluorine-18 is no less a remarkable and versatile positron-emitting radionuclide for PET imaging. Decay of fluorine-18 is largely by positron (β^+) emission (97%), with a relatively low energy (maximum β^+ energy of 0.635 MeV), and thus the emitted particle has a short mean range (2.39 mm in water) making it an attractive radioisotope for localization measurements requiring high-resolution PET imaging. The moderate half-life of fluorine-18 ($t_{1/2}$ 109.7 min) allows for considerable latitude in the synthesis of radiopharmaceuticals.

Historically, a variety of nuclear reactor, linear-accelerator and cyclotron methods have been used to produce fluorine-18. Reactor produced fluorine-18 is plagued by a complicated method requiring a two-step process involving fast neutron bombardment of a solid lithium-6 target that would produce tritons to drive the $^{16}\text{O}(t, n)^{18}\text{F}$ nuclear reaction. In addition to the complicated targetry involved here, there are often issues of tritium contamination in the workplace. Today, cyclotron production is clearly the method of choice owing to higher production yields and fewer radioactive contaminants. For all practical purposes nearly all fluorine-18 is currently produced using the single nuclear reaction $^{18}\text{O}(p, n)^{18}\text{F}$ on an oxygen-18 enriched liquid water target--the majority of the radionuclide is isolated as the [^{18}F]-fluoride ion in an aqueous solution and can be rendered suitable for labeling *via* nucleophilic substitution reactions. Alternatively, the radionuclide can be manipulated either through changes in targetry, or through post-irradiation chemistry to produce [^{18}F]- F_2 which is suitable for electrophilic substitution reactions.

In aqueous form [^{18}F]-fluoride ion is not very reactive, but through simple manipulations, its reactivity toward organic molecules and solubility in nonpolar organic solvents can be enhanced making labeling useful. To do this, the [^{18}F]fluoride ion must be accompanied by a positively charged counterion. Unfortunately, the metal ions that elute the target after irradiation of ^{18}O -enriched

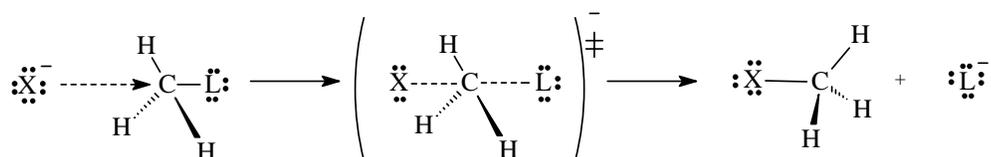
liquid water are not of sufficient composition or concentration to provide a source of reactive fluoride after evaporation of that water. This problem is very effectively solved by the addition of a cationic counterion prior to the evaporative drying step (see procedures). Three types of counterions can be used for this purpose: (1) large alkali metal ions such as Rb^{+2} or Cs^{+2} whose salts do offer modest solubility in organic solvents; (2) K^{+} ion complexed by a cryptand such as the crown ether, Kryptofix 2.2.2-- the limited solubility of K^{+} in nonpolar organic solvents as compared with larger metal ions necessitates complexing it within crown ether; or (3) tetraalkylammonium salts.



Past practices using ^{18}F -fluoride ion have shown that it can efficiently displace a variety of leaving groups (notably halide atoms) that are attached to an organic substrate—particularly when that substrate is an aromatic ring like benzene. Furthermore, this displacement is often enhanced when there are strong electron-withdrawing groups attached ortho or para to the particular site under attack by the radioactive nucleophile.

The mechanism for ^{18}F nucleophilic aromatic substitution is somewhat interesting. It cannot proceed by a bimolecular $\text{S}_{\text{N}}2$ mechanism because these substituted aryl compounds cannot achieve the correct geometry for back-side attack characteristic of this mechanism.

$\text{S}_{\text{N}}2$ Mechanism

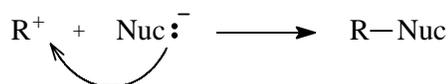


Furthermore, the reaction cannot precede by a unimolecular $\text{S}_{\text{N}}1$ mechanism as the reaction rate would then be dependent only on the concentration of the nucleophile ($^{18}\text{F}^-$). Labeling reactions carried out under no-carrier-added conditions (at high specific activity) would have minute amounts of radiotracer relative to the substrate. Hence, the reaction would be very inefficient if driven by an $\text{S}_{\text{N}}1$ mechanism.

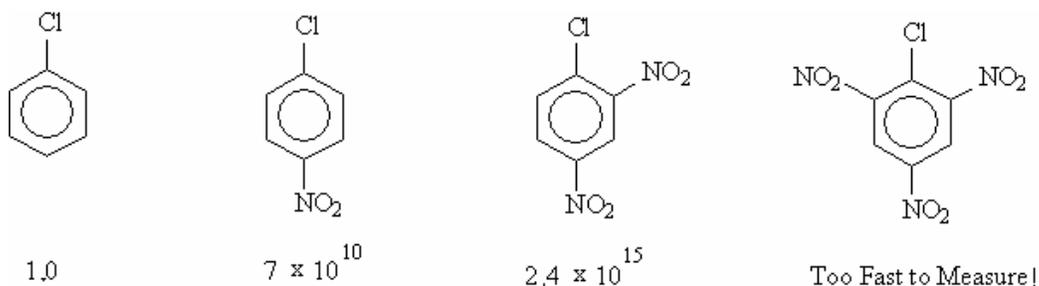
Step 1: carbocation formation (slow)



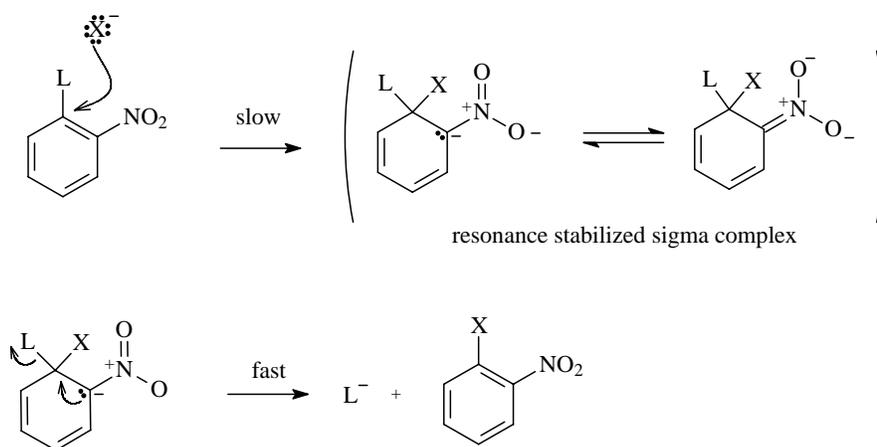
Step 2: nucleophilic attack (fast)



One thing noted with ^{18}F labeling on aromatic rings is that electron-withdrawing substituents tend to activate the ring toward nucleophilic aromatic substitution, suggesting that the transition state develops a negative charge which translates back on the ring. **(In fact, nucleophilic aromatic substitutions are difficult to carry out in general, unless there is at least one powerful electron-withdrawing group on the ring.)** Consider the following reaction rates for nucleophilic substitution on various aryl chlorides. The over-all rate of reaction increases dramatically as more nitro groups are added to the aromatic ring in the ortho and para positions. These additional nitro groups can better stabilize the transition-state.



One mechanism that best explains the nature of these labeling reactions involves addition-elimination.



In this process, both leaving group “L” and nucleophile “X” remain bound to the ring in the transition state. Note how the negative charge is drawn by the nitro electron withdrawing group which helps to stabilize the transition state. Three factors clearly play a role in the efficiency of this reaction: (1) nucleophilicity of X; (2) nature of the leaving group (L) which is defined by its size (steric hindrance) and C-L bond strength; and (3) nature of other ring substituents which will affect the stability of the transition state.

Today you will explore two features of this reaction including: (1) temperature, and (2) nature of leaving group. You will be using $[^{18}\text{F}]$ -fluoride ion as our

nucleophile for testing reaction efficiency. **Note—you will need to compile and share your data with other teams for final write-up and interpretation.**

An essential element toward the success of today's experiments is your ability to measure the extent of reaction using radio thin layer chromatography (radio TLC). You will be using a Bioscan AR-2000 radio TLC scanner for this purpose. The scanner uses a gas-filled proportional counter, which can detect all beta and gamma emitting isotopes. An entire TLC lane can be imaged in less than one minute, which is usually sufficient to analyze and calculate the purity of a compound. The system utilizes WinScan instrument control and data acquisition software. Results are presented as chromatograms. Quantitation of peaks is automatically performed and a report showing the method used, chromatogram, and percent of total activity for each peak is provided.



MATERIALS AND APPARATUS

1. 3 Vac container tubes (will be used for reaction vessels)
2. Temperature controlled oil bath (each team has one oil bath).
3. Argon gas for dry down step.
4. Potassium carbonate (K_2CO_3)—4 mg per reaction.
5. Kryptofix 2.2.2—20 mg per reaction.
6. Acetonitrile—approximately 10 mL per reaction.
7. Dimethyl sulfoxide (DMSO) —300 μ L per reaction.
8. Small glass screw cap vials (3) for making substrate solution.
9. Silica TLC plates (at least 3).
10. TLC pipette tips.
11. TLC solvent (1:1 (v/v) hexane:ethyl acetate).
12. Ruler and pencil for marking TLC plates.
13. TLC glass developing tank.

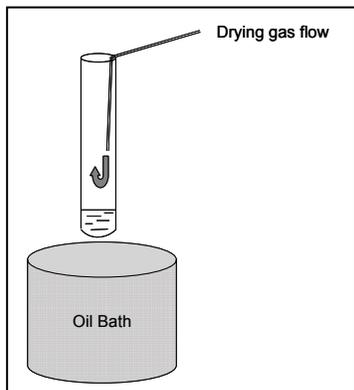
14. Bioscan radio TLC scanner.
15. Labeling substrates.
 - Part 1: Temperature effect using 1,2-dinitrobenzene (**All Teams**)
 - Part 2: Assessing leaving group effects using the following substrates:
 - Team 1: 1-fluoro-2-nitrobenzene
 - Team 2: 1-chloro-2-nitrobenzene
 - Team 3: 1-bromo-2-nitrobenzene
 - Team 4: 1-iodo-2-nitrobenzene
16. Microcurie source of [¹⁸F]fluoride ion (to be dispensed by Ferrieri).
17. 100 μL pipettor (for dispensing 50 μL volumes of water per reaction).
18. 1000 μL pipettor.
19. Vortex mixers (two units shared by all).
20. Analytical balances (two units shared by all).
21. Weighing paper for measuring reactants.
22. Two large 500 mL beakers (used as waste sites for syringes, pipette tips and anything else that will accumulate during this lab).
23. Black light for measuring UV absorbance on TLC plates.
24. Stopwatch
25. Forceps

PROCEDURES

Part I: Measuring Effect of Temperature on Reaction Rate.

Each team will weigh out approximately 4 mg of K₂CO₃, 20 mg of Kryptofix 222 and place these reagents into a new Vac container tube. Dispense 50 μL of distilled water into the tube and vortex for 2-3 minutes. Ensure all the carbonate salt has dissolved before proceeding. **Add a miniature magnetic stirring bar to the tube.** Fix the open Vac container tube (using a ring clamp and stand or support rail) above the oil bath (set the oil bath at 120°C for the initial dry down step). Attach a 6 inch stainless syringe needle onto the argon (or nitrogen gas) flow line (this will have already been set up by your TA). Ensure that you have adequate flow of gas when the valve is open. (An easy test is to dip the needle end into a vial of acetone and check for bubbles—do this at the beginning of the lab when there is no radioactivity in your dispersibles workzone.) Place the end of the syringe needle into the Vac

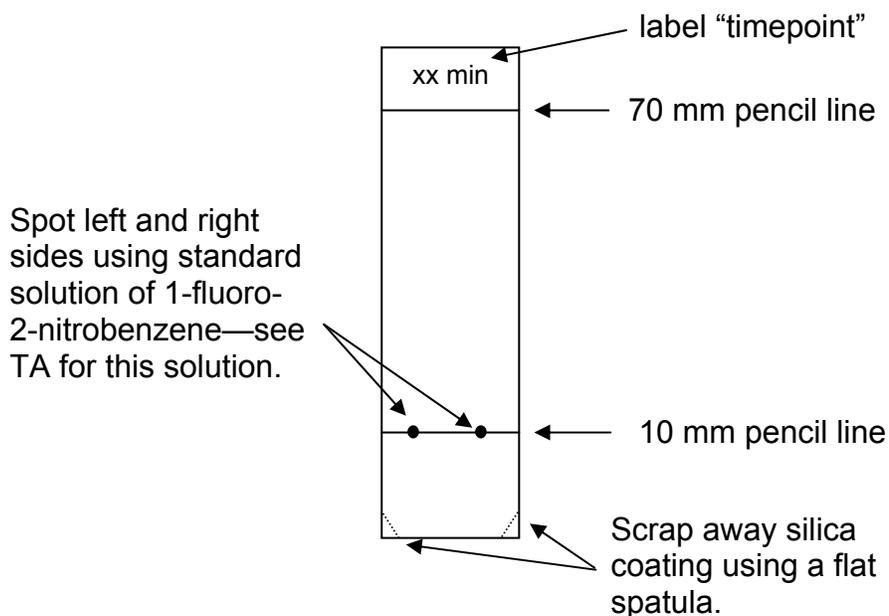
container tube so that the end of the needle rests about half way up. (See diagram) Using the 1000 μL pipettor dispense 300 μL of acetonitrile into the test tube. Ask the TA to dispense a microliter volume of [^{18}F]-fluoride ion into the test tube. **This amount should allow for about 20-30 μCi of radioactivity per reaction.** You do not need to know the absolute amount of radioactivity dispensed as we will be using radio TLC to measure the extent of chemical reaction.



Lower the test tube into the oil bath (set to 120°C) with the drying gas flowing and monitor drying. Once dry add 1 mL of acetonitrile using a disposable 1 mL syringe. (Remember to place the used syringe into the waste beaker located near to where you are working.) The acetonitrile facilitates removal of water (by forming an azeotropic mixture) entrained in the cryptand complex. Continue drying. Repeat this step 1 more time. At this point remove the Vac Tube from the oil bath and readjust the oil bath to the desired reaction temperature. (**Team 1, 30° C; Team 2, 50° C; Team 3, 70° C; Team 4, 90° C**)

If necessary, ask your TA to add bits of dry ice to the oil to speed the cooling process.

During temperature re-equilibration you will need to prepare three (3) TLC plates. Using the following procedure. Using a metric ruler and pencil draw lines across your silica TLC plate at 10 mm (origin) and 70 mm (solvent front).

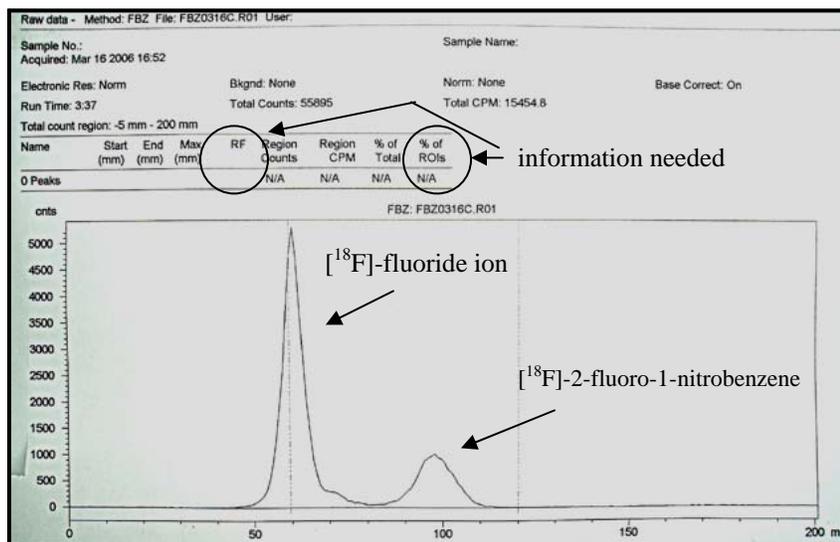


Using a pencil mark two spots on the 10 mm line. Using the flat edge of a spatula scrap away some of the silica coating as shown in figure—this helps create a straight solvent front as it rises up the plate. Once prepared, place your TLC plates in an orderly fashion into the stainless steel trays marked as radioactive material. Using a glass microspotter, spot each pencil mark 2x using a standard solution of 1-fluoro-2-nitrobenzene that your TA will provide. Allow spots to dry. Prepare your

TLC developing tank by pouring approximately 10 mL of 1:1 mixture (v/v) of hexane:ethyl acetate (TA will provide this solvent) into the bottom of it—note, you should not exceed 10 mm height with solvent. Swirl the contents in the tank with its glass cover in place for 30 seconds to allow adequate equilibration of solvent with the chamber.

Next weigh out 2 mg of 1, 2-dinitrobenzene and place it into a small glass vial. Add 300 μ L of DMSO to the vial and vortex for 3 minutes. Using a 1 mL disposable syringe (equipped with a 22 gauge “yellow” needle) draw up contents and dispense into the Vac container tube containing your radioactive [18 F]fluoride/kryptofix mixture. Lower the tube into the heated oil (at desired temperature) and allow contents to react for up to 15 minutes (use your stopwatch to time down). **At 5 minute intervals you will dip a new glass TLC spotter into the reaction solution and spot the right-side of the appropriate TLC silica plate 2-times.** Bring the stainless steel tray holding your sample TLC plate close to where you are sampling—never remove the spotter from the solution unless you have your tray and plate on hand. Dispose of your spotter in the appropriate radiation waste receptacle marked in your dispersibles zone. **The person doing the spotting should change his/her second layer of gloves at this point.**

Using forceps, carefully place your TLC plate (face up) into the TLC developing tank resting its top edge along one side of the tank at a slight angle—try not to disturb the solvent in the bottom when you do this. Carefully monitor as the plate develops to ensure that the solvent front does not travel beyond the 70 mm pencil line. Once developed, carefully remove the plate and lightly evaporate the solvent using a hot air gun. Place the TLC plate onto the Bioscan radio TLC scanner lining up the radioactive spot with the tab. Also ensure that you have lined up the solvent origin line with the mark on the scanner’s bed. You will be using the “FBZ method” for scanning your plate. Your TA’s and PET staff will be on hand to assist in running your plates. The scanner will count for 5 minutes before printing out a copy of the axial distribution of radioactivity and final report. You can stop the counting prior to the 5 minute period if you see 5000 cts. The program will automatically print your scan and report at termination. See example below of a TLC scan and report.



You will need to record R_f and percent of product from this report. Also, save this report as you will need to make xerox copies for fellow team members to use in their final write-up.

Place the TLC plate under the black light and record using a pencil where you see spots along the plate. It should be easy to identify the product peak as the left side of your plate was spotted only with authentic fluoronitrobenzene (FNBz). You should also see a rather large spot from the starting material (dinitrobenzene). Calculate the R_f for FNBz and compare with the R_f for [^{18}F]FNBz recorded by the radio TLC scanner. Use the equation below for calculating R_f :

$$R_f = [\text{distance (mm) to center of product spot}] \div [\text{distance (mm) of solvent front}]$$

Record in your notebook the reaction temperature, product yield, product R_f , and the sampling time. Also, compile a list of temperatures and reaction yields from the other teams.

Before continuing to Part II ensure any sources of radioactive material are placed in the radiological waste receptacle. Rubber stopper your reaction test tube and place that in the receptacle as well.

Part II: Measuring the Effect of Leaving Group on Reaction Rate.

Before continuing with Part II, ensure that all radioactive waste from Part I is disposed of in the appropriate radioactive waste receptacle, and check your workzone using the GM frisker to ensure there is no dispersed radioactivity.

For Part II of this lab you will explore the effects of leaving group on nucleophilic aromatic substitution reactions using [^{18}F]fluoride ion as the nucleophile. Each team will be issued a new substrate as assigned below:

Team 1: 1-fluoro-2-nitrobenzene

Team 2: 1-chloro-2-nitrobenzene

Team 3: 1-bromo-2-nitrobenzene

Team 4: 1-iodo-2-nitrobenzene

You will use the **same procedures** as described in Part I of this lab for carrying out your reactions. The drying step will be carried out at 120°C . After drying adjust the oil bath to 90°C which will be your reaction temperature. Carry out the reaction for 15 minutes and sample the crude reaction mixture at the end of this period for radio TLC workup. You will be developing only one TLC plate. **Again, data must be compiled between the teams for interpretations of leaving group effects in your final write-up.**

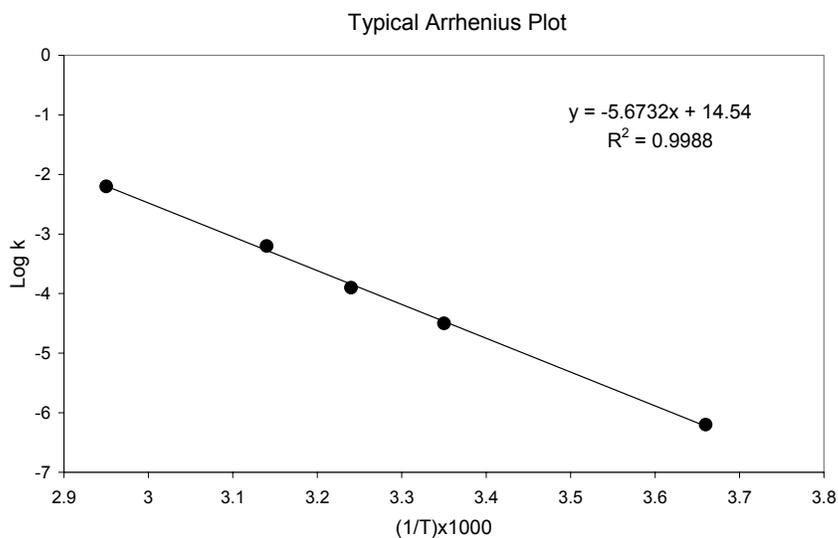
DATA ANALYSIS

Arrhenius Kinetics: If the temperature range is not too great, the dependence of rate constants on temperature can usually be represented by an empirical equation proposed by Arrhenius, $k = Ae^{-E_a/RT}$ where A is the pre-exponential factor and E_a is the activation energy of reaction and T is the reaction temperature in Kelvin. This equation may be written in logarithmic form as:

$$\log k = \frac{E_a}{2.303 R} \left(\frac{1}{T} \right) + \log A$$

If you assume pseudo first-order kinetics (why can you assume this when working with relatively high specific activity ^{18}F ?), the rate constant k can be related to the amount of ^{18}F by the relation by the following relationships below. Construct a graph in Excel to calculate reaction rates by plotting the product radiochemical yield on the Y-axis versus the reaction time (0, 5, 10, 15 minute) on the X-axis. Fit the data using Linear Regression analysis and display the equation and R^2 fit on the graph. Calculate the rate, k, for each temperature from the slope of the line.

Once you have rates calculated at each temperature construct an Arrhenius plot in Excel of Log k versus $[(1/T) \times 1000]$ and fit the data using Linear Regression analysis. The plot should look something like the figure below.



The slope of the graph is -5673 deg, and E_a has the value of:

$-2.3 \times R \times (\text{slope}) = 25,926.2 \text{ cal/mol}$ or 25.9 kcal/mol rounded off.

Calculate the activation energy for the reaction (in kcal/mol) from the slope of your graph like in the example above. Remember to use $R = 1.987 \text{ cal/deg}\cdot\text{mol}$ and your temperature units need to be in degrees Kelvin when you construct your plot.

Leaving Group Effects: In Part II of this lab you explored the effect of leaving group on reaction rate. From the compiled data you have from the other teams, make a plot of the radiochemical yield versus electronegativity using the following data for aid.

	R-I	R-Br	R-Cl	R-F	R-NO ₂
C-X(kcal)	53	69	82	109	174
Electronegativity	2.3	2.8	3.0	4.0	4.2

POST LAB DATA DELIVERABLES

Notebook

- Include a brief statement of the lab's objectives.
- Include a sketch of your experimental setup.
- Include a brief (1 paragraph) description of the procedure showing reactions—hand-drawn reactions are fine.
- Include printouts of your individual team's radio TLC plots and include product labels and R_f values on both authentic product and labeled product.
- Make up a table of the compiled radiochemical yields from the combined effort of all 4 teams—this table should have results from the temperature effect and leaving group effect studies.
- Include any observations from the lab that may have impacted your results.

Lab Report

- Include a 1-page summary of what this lab was about (key elements we are looking for include title, date, lab partners, statement of objectives a brief statement on how these objectives were met through a description of the basic principles being demonstrated).
- Include all plots (4) of product radiochemical yield versus time for the four different temperatures with data fitted by Linear Regression Analysis and the equation and R^2 fit displayed. Your table of yields compiled in your notebook need not be included here, but you are asked to plot the yield data from the other teams. Note you can include all four lines on a single graph.

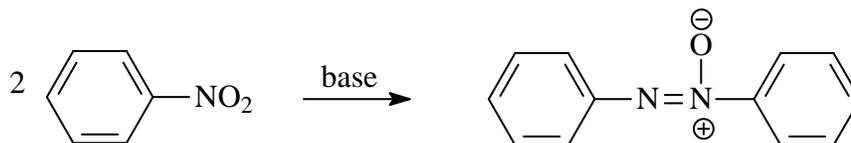
- Include your Arrhenius plot labeled showing equation and best fit. Calculate an activation energy for the denitration reaction.
- Include a plot of radiochemical yield versus leaving group electronegativity.
- Include a discussion of trends by answering the questions below.
- Include a summary statement noting challenges of radiofluorination chemistry based on your experience in the lab.

Questions for Discussion

1. Comment on the behavior of the reaction kinetics in today's experiment relative to temperature. Is it what you expected for Arrhenius behavior?
2. Consider the fact that gaseous fluoride anion is a potent nucleophile with activation energies for nucleophilic aromatic substitution with dinitrobenzene on the order of 3.5 kcal/mol. Comment on the nucleophilicity of your [¹⁸F]-fluoride / krytoxfix system relative to this information.
3. For the simple substitution reaction tested in Part I discuss why you would not expect to see further labeling of the substrate. Assume that the specific activity of your [¹⁸F]-fluoride ion is 1 Ci/μmol and that your reaction was carried out using approximately 10 μCi of material. Show calculations.
4. A student mistakenly used KHSO₃ instead of K₂CO₃ during the initial dry down. When he went to spot his TLC plate after his reaction he discovered there was no radioactivity in his sample. Discuss what may have happened.
5. Consider the data presented in the table below where [¹⁸F]-fluoride ion reactions with 1,2-dinitrobenzene were tested at higher reaction temperatures.

¹⁸ F-FNBz	
Radiochemical Yield (%)	Reaction Temperature (C)
74	120
53	140
45	150
26	165
8	175

Consider the fact that under basic conditions nitrobenzene will form azoxybenzene.



Write a mechanism that would account for your loss of [¹⁸F]-1-fluoro-2-nitrobenzene. Also consider that azoxybenzene can be reduced and cleaved to its respective anilines by refluxing in with Zn metal. Write a possible reaction describing the fate of ¹⁸F under these circumstances. Discuss a corrective action, **other than reaction temperature**, that would allow you to minimize this secondary reaction in your labeling scheme.

Additionally, radiolabeled anilines are extremely useful as building blocks for larger biomolecules of interest. Discuss why it is not possible to carry out a direct radiosynthesis of ortho or para [¹⁸F]-fluoroaniline using [¹⁸F]-fluoride ion.

5. Discuss the implications of any systematic trends you observed due to nature of the leaving group. Which ligand makes the best leaving group? Is this what you would expect for classical S_N2 substitutions?
6. Discuss why it is not practical in PET to use a fluoro-substituted substrate for radiolabeling with ¹⁸F.

References

“Handbook of Radiopharmaceuticals: Radiochemistry and Applications,” Welch M.J. and Redvanly C.S. (eds.) John Wiley & Sons, Ltd., Chichester, England (2003), Chapters 6 & 7. (See NCSS reference shelf)

Appendix 1: Counting Statistics

Here are some examples of how to use this type of statistics to help determine the uncertainty in your data or to determine its accuracy and precision.

1. Standard Deviation (s) for total count using Gaussian Distribution

Gross sample count rate: $R = 1000$ cpm

total count time = 10 minutes

total count: $n = 1000$ cpm \times 10 min = 10,000 counts

$s = \sqrt{n} = \sqrt{10,000} = \pm 100$

Therefore:

counts $\pm s = 10,000 \pm 100$ counts or 9900 - 10,100 counts range for 68% probability.

Consequently, if this sample is counted 100 times, 68 times the total count will be between the limits of 9900 - 10,100 counts, and 32 times, beyond these limits.

2. Standard Deviation (s_R) in the counting rate, (R):

$$s_R = \frac{\sqrt{n}}{t} = \sqrt{\frac{R}{t}} \quad (1)$$

s_R for the previous example:

$$\frac{\sqrt{10,000}}{10} = \frac{100}{10} = \pm 10 \text{cpm}$$

3. Confidence Levels and K:

The confidence level expresses the % probability of the true value occurring within certain limits.

$$K = \text{number of standard deviations} = \left| \frac{\langle n \rangle - n}{\sigma} \right|$$

Table 1. Table of Constants of Relative Error

Confidence Level	K	
50.00	0.6745	Probable Error
68.27	1.0000	Standard Deviation (1s)
90.00	1.6449	Nine tenths Error
95.00	1.9600	Ninety-five hundredths error
95.45	2.0000	2s
99.00	2.5758	Ninety-nine hundredths error
99.73	3.0000	3s
99.9937	4.0000	4s

To determine the limits, $\pm \Delta n$ corresponding to a certain confidence level for a total count of n :

$$\Delta n = K_i \sigma = K_i \sqrt{n} \quad (2)$$

where K_i is the K corresponding to the particular confidence level.

Example: For a total count $n = 10,000$, for probable error (or 50% confidence level):

$$\Delta n = 0.6745 \sqrt{n} = 0.6745 \sqrt{10,000} = \pm 67$$

Work out for yourself that for 95% confidence level, $\Delta n = \pm 196$

4. Error in Total Counting System

Uncertainty in Total Counting System where Total Count is the sample count +

background count and therefore, the sample count is the total count - background count:

4.1 Standard Deviation for Total Sample Count:

$$\sigma_s = \sqrt{\sigma_i^2 + \sigma_b^2} = \sqrt{n_t + n_b} \quad (3)$$

When $\sigma_b \ll \sigma_t$,

$$\sigma_s \approx \sqrt{\sigma_i^2} \approx \sigma_i \quad (4)$$

4.2 Standard Deviation for Sample Counting Rate Alone:

$$\sigma_{R_s} = \sqrt{\sigma_{R_t}^2 + \sigma_{R_b}^2} = \sqrt{\frac{R_t}{t_t} + \frac{R_b}{t_b}} \quad (5)$$

When $\sigma_{R_b} \ll \sigma_{R_t}$, $\sigma_{R_s} \approx \sqrt{\sigma_{R_t}^2} \approx \sigma_{R_t}$

4.3 Optimization of counting conditions: $\frac{t_b}{t_t} = \sqrt{\frac{R_b}{R_t}} \quad (6)$

Example: Suppose you are using a counter with a background count rate (R_b) of 50 cpm and are only allotted one hour to count your sample, which has a gross count rate (R_t) of 2000 cpm. How should you proportion your allotted time between sample and background for minimum error?

$$R_b = 50 \text{ cpm}$$

$$R_t = 2000 \text{ cpm}$$

$$t_t = 60 \text{ min}$$

$$\frac{t_b}{t_t} = \sqrt{\frac{R_b}{R_t}} = \sqrt{\frac{50\text{cpm}}{2000\text{cpm}}} = 0.158$$

$$t_b/60 \text{ min} = 0.158 \text{ and } t_b = 0.158 \times 60 \text{ min} = 9.48 \text{ min}$$

$$t_t = 60 \text{ min} - 9.48 \text{ min} = 50.52 \text{ min}$$

5. Calculation for preset time (pst) or preset count (psc) for a predetermined accuracy:

$$\text{pst} = \frac{K^2 \times 10^4}{F^2} * \frac{1}{R_s} \left[1 + \frac{2R_b}{R_s} \right] \quad (7a,b)$$

$$\text{psc} = R_t * \frac{K^2 \times 10^4}{F^2} * \frac{1}{R_s} \left[1 + \frac{2R_b}{R_s} \right]$$

where F = desired error as a percentage.

Example: Suppose you had a total count rate of 600 cpm and the background rate was 50 cpm. How long would you have to count your sample to obtain 2% accuracy at 99% confidence level?

$$R_s = R_t - R_b = 600 \text{ cpm} - 50 \text{ cpm} = 550 \text{ cpm}$$

K (for 99% confidence level from Table 1) = 2.5758 and F = 2

$$\text{pst} = \frac{2.5758^2 \times 10^4}{2^2} \times \frac{1}{550} \left[1 + \frac{2 \times 50}{550} \right] = 35.6 \text{ min}$$

Suppose you counted the same sample to a preset count of 36,000, what would your percent accuracy be at a 95% confidence level?

$$\text{psc} = R_t * \frac{K^2 \times 10^4}{F^2} * \frac{1}{R_s} \left[1 + \frac{2R_b}{R_s} \right]$$

$$F = \sqrt{R_t * \frac{K^2 \times 10^4}{\text{psc}} * \frac{1}{R_s} \left[1 + \frac{2R_b}{R_s} \right]}$$

K=1.96 for 95% confidence level

$$F = \sqrt{600\text{cpm} * \frac{1.96^2 \times 10^4}{36,000} * \frac{1}{550} \left[1 + \frac{2 \times 50}{550} \right]} = 1.17\%$$

REFERENCE

P. R. Bevington, D. K. Robinson, P. Bevington, Data Reduction and Error Analysis for the Physical Sciences, McGraw Hill, 3rd Ed. (2002).

Appendix 2a.

Operation Instructions for the Spectech GM Counters

1. There is no way to tell if the high voltage is on or not on these detectors when they are powered off. To prevent problems, always turn HV to zero before turning the detector off.
2. Insert the BNC cable from the detector to the back of the counter. To install a BNC, line up the slots, then turn the connector a quarter turn to lock the connection.
3. Plug in the power adapter into the back of the counter, and then plug the adapter into the wall socket.
4. Turn the system on (switch is on the back of the counter). Allow a minute for the system to warm up.
5. This system has several options on the dial ranging from counts to high voltage. Turning the dial selects the option, and the results are displayed on the LCD screen. Pay careful attention to the dial when you are observing or recording the screen output. Red LEDs on the front of the unit also help to indicate the output.
6. Place a beta source on shelf 3. *Note: It matters which side of the source faces the detector. If you do not record any activity, try turning the source over.*
7. Set an arbitrarily long count time: Turn the dial to “time” and select a time. The units are in seconds.
8. Start counting your source: Turn the dial to “count”. Press the “count” button on the front of the unit (the LED should light up).
9. Turn the voltage to the proper setting: Turn the dial to “high voltage” and use the “up” and “down” keys to adjust the voltage. If you do not know the voltage to use, generate a voltage response curve.
10. When high voltage is properly adjusted, press the “stop” button. Press the “reset” button to zero the instrument. The counter is now ready for operation. You must press “stop” in order to “reset”.
11. Perform your experiments.
12. Turn down the high voltage to zero: Turn the dial to “high voltage” and push the “down” button until the readout displays zero.
13. Turn off the system.

Appendix 2b.

Operation Instructions for Model 575 GM Detectors

1. **Make sure that the high voltage is turned to zero before turning on the detector.**
2. Plug the line cord into an outlet and ensure that the Geiger tube is connected to the GM input jack.
3. Turn the system on (switch is on front of the detector) and allow a 1 minute warm-up.
4. Place a beta source on a shelf 2. *Note: It matters which side of the source faces the detector. If you do not record any activity, try turning the source over.*
5. Set the count time to some arbitrarily long time and start counting by pressing the **COUNT** switch (count length is determined using the two dials on the right side of the front of the instrument). *Note: The unit count time is in minutes.*
6. Turn the voltage to the proper setting. If you do not know which voltage to use, generate a voltage response curve.
7. When the high voltage is properly adjusted, press the **STOP** switch. Press the **RESET** switch to zero the instrument. The scaler is now ready for operation.
8. Perform your experiments.
9. Turn off the high voltage
10. Turn off the system.

Appendix 3: Common Pure Beta Standards

Nuclide	Half-life	Fractional Abundance (%)	$E_{\beta\max}$ (keV)
^{14}C	5730 y	100	157
^{147}Pm	2.627	100	224
^{33}P	24.4 d	100	248
^{36}Cl	3.0×10^5 y.78 y	98.1	709
^{204}Tl	3.78 y	97.1	763
^{32}P	14.3 d	100	1710.5
^{90}Y	64.1 h	100	2282.0

Appendix 4: Common Pure Gamma Standards*

Nuclide	Half-life	Fractional Abundance (%)	E_{γ} (keV)
^{57}Co	271.8 d	9.16 85.60 10.68	14.4 122.1 136.5
^{109}Cd	462.0 d	3.79	88.0
^{65}Zn	243.8 d	50.8	1115.5 511 (due to weak β^+ - 1.46%)
^{54}Mn	312.1 d	100	834.8

Appendix 5: Common Alpha Standards

Nuclide	Half-life	Fractional Abundance (%)	E_α (MeV)
^{210}Po	138.38 d	99+	3.72

*see Appendix 4 for other common β/γ standards

Appendix 6: Common Gamma Standards*

Nuclide	Half-life	Fractional Abundance (%)	E_γ (keV)
^{241}Am	432.7 y	36.9	59.5
^{57}Co	271.8 d	9.16 85.60 10.68	14.4 122.1 136.5
^{109}Cd	462.0 d	3.79	88.0
^{133}Ba	10.7 y	32.7 7.3 18.62 62.27 8.84	81.0 276.3 302.7 355.9 383.7
^{137}Cs	30.07 y	84.62	661.7
^{65}Zn	243.8 d	50.8	1115.5 511 (due to weak b^+ - 1.46%)
^{60}Co	5.271 y	99.86 99.98	1173.5 1332.5
^{22}Na	2.604 y		1274.5 511
^{54}Mn	312.1 d	100	834.8

^{241}Am decays also with α - emission, ^{133}Ba , ^{137}Cs , ^{60}Co , and ^{22}Na decay also with β -emission.

Appendix 7: Brief Oscilloscope Tutorial

INTRODUCTION

The oscilloscope is your friend. Really! I find that students are more intimidated by the oscilloscope than any other equipment in the laboratory so this was written to help you out. If you read this before the first laboratory, An Introduction to Pulse Instrumentation, you won't approach the oscilloscope with either a glazed expression or a desire to randomly turn dials to see what happens (though sometimes that works!). In any case, this course assumes no previous knowledge. Most of this writeup is simply cut directly from the website: <http://www.cs.tcd.ie/courses/baict/bac/jf/labs/scope/oscilloscope>. Another good

site to find out more about scopes is:

http://www.tek.com/Measurement/App_Notes/XYZs/03W_8605_2.pdf.

What is an oscilloscope, what can you do with it, and how does it work? This section answers these fundamental questions. The oscilloscope is basically a graph-displaying device - it draws a graph of an electrical signal. In most applications the graph shows how signals change over time: the vertical (Y) axis represents voltage and the horizontal (X) axis represents time. The intensity or brightness of the display is sometimes called the Z axis. (See Figure 1).

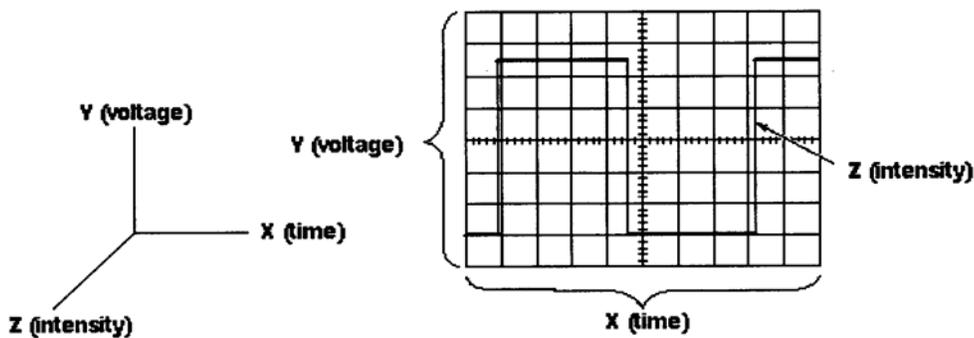


Figure 1: X, Y, and Z Components of a Displayed Waveform

This simple graph can tell you many things about a signal. Here are a few:

- You can determine the time and voltage values of a signal.
- You can calculate the frequency of an oscillating signal.
- You can see the "moving parts" of a circuit represented by the signal.

- You can tell if a malfunctioning component is distorting the signal (very helpful when debugging your detector system).
- You can find out how much of a signal is direct current (DC) or alternating current (AC).
- You can tell how much of the signal is noise and whether the noise is changing with time.

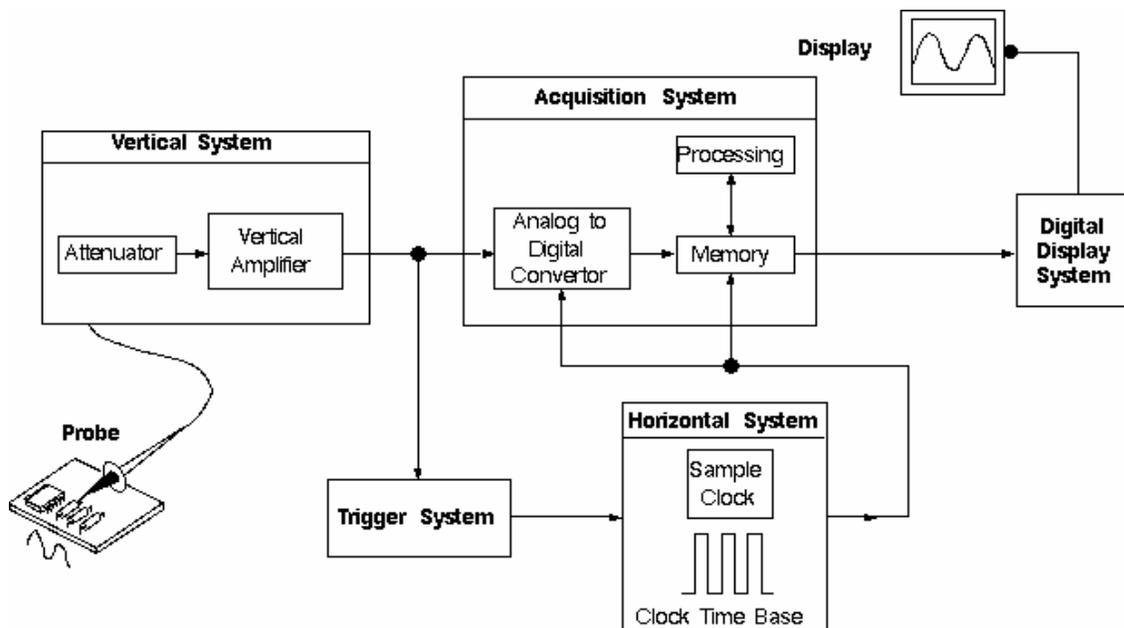
Oscilloscopes are used by everyone from television repair technicians to physicists. They are indispensable for anyone designing or repairing electronic equipment. The usefulness of an oscilloscope is not limited to the world of electronics. With the proper transducer, an oscilloscope can measure all kinds of phenomena. A transducer is a device that creates electrical signals in response to physical stimuli, such as sound, mechanical stress, pressure, light, or heat. For example, a microphone is a transducer. An automotive engineer uses an oscilloscope to measure engine vibrations. A medical researcher uses an oscilloscope to measure brain waves. The possibilities are endless.

The oscilloscopes we will use in this course are digital oscilloscopes (Figure 2).



A schematic is shown in Figure 3. The input to a digital scope goes to a very high speed analog to digital converter which samples the input waveform up to as many as 1.25×10^9 samples per second (for our scopes). There is thus a set of x,y coordinates for every sampling point. Each x,y pair corresponds to a particular pixel on the LCD screen and the waveform is thus displayed on the LCD screen with points so close together that it appears as a continuous waveform. The scope has two distinct modes of operation. In x-y mode the user supplies both the horizontal (x) and the vertical (y) deflection signals through input connectors located on the front of the scope, so that what appears on the

Fig. 3: Digital Oscilloscope Schematic.



screen is a plot of y vs x . In time base mode the time samples are drawn across the screen from left to right at a constant speed (which the user can select) while the vertical deflection is generated from an input signal supplied by the user. This makes it possible to view the input signal directly as a function of time.

OSCILLOSCOPE FRONT PANEL

Take a look at the oscilloscope screen and you will see a grid pattern on it (like Figure 1 above). Usually these squares are 1 cm square. An oscilloscope looks a lot like a small television set, except that it has this grid drawn on its screen and it has more controls than a television. The front panel of an oscilloscope normally has control sections divided into **Vertical**, **Horizontal**, and **Trigger** sections. There are also display controls and input connectors. Your oscilloscopes display on the screen how many volts each vertical division represents and how many seconds each horizontal division represents. The oscilloscopes also have 0%, 10%, 90%, and 100% markings on the graticule (see Figure 1) to help make rise time measurements, described later.

On all oscilloscopes, the controls are grouped according to three functions: 1) Vertical motion controls (voltage, y axis); 2) Horizontal motion controls (time, x axis); and 3) Control of the time base circuits (trigger).

Vertical Controls: Use the vertical controls to position and amplify the waveform vertically. Your oscilloscope also has controls for setting the input coupling and other signal conditioning, described in this section. The vertical position control lets you move the waveform up or down to exactly where you want it on the screen (**position knob** under the Vertical heading on your scope). The actual volt units appear under the x -axis to the left. The volts per division (**Volts/div knob**) setting varies the size of the waveform on the screen. A good

general purpose oscilloscope can accurately display signal levels from about 4 millivolts to 40 volts.

The volts/div setting is a scale factor. For example, if the volts/div setting is 5 volts, then each of the eight vertical divisions represents 5 volts and the entire screen can show 40 volts from bottom to top (assuming a graticule with eight major divisions). If the setting is 0.5 volts/div, the screen can display 4 volts from bottom to top, and so on. The maximum voltage you can display on the screen is the volts/div setting times the number of vertical divisions. Note: Your oscilloscope has a **Probe** button just to the right of the screen. Select 1X for your measurements. Selecting 10x magnifies the scale by a factor of ten.

Often the volts/div scale has either a variable gain or a fine gain control for scaling a displayed signal to a certain number of divisions. Use this control to take rise time measurements.

Horizontal Controls: Use the horizontal controls to position and scale the waveform horizontally. The horizontal position control (**position** knob under the Horizontal heading on your oscilloscope) moves the waveform from left and right to exactly where you want it on the screen.

The seconds per division (**sec/div** knob) setting lets you select the rate at which the waveform is drawn across the screen (also known as the time base setting or sweep speed). The actual time units are displayed below the x-axis in the bottom center on the screen. This setting is a scale factor. For example, if the setting is 1 ms, each horizontal division represents 1 ms and the total screen width represents 10 ms (ten divisions). Changing the sec/div setting lets you look at longer or shorter time intervals of the input signal.

As with the vertical volts/div scale, the horizontal sec/div scale may have variable timing, allowing you to set the horizontal time scale in between the discrete settings.

Trigger Controls: Figure 4 (next page) shows you why the trigger is important but what is it?

An oscilloscope's trigger function synchronizes the horizontal sweep at the correct point of the signal, essential for clear signal characterization. Trigger controls allow you to stabilize repetitive waveforms and capture single-shot waveforms. The trigger makes repetitive waveforms appear static on the oscilloscope display by repeatedly displaying the same portion of the input signal. Imagine the jumble on the screen that would result if each sweep started at a different place on the signal, as illustrated in Figure 4.

Learning how to set and adjust trigger control is one of the more

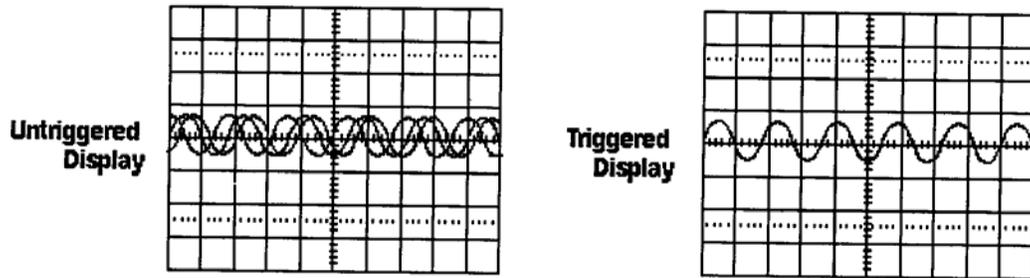


Fig. 4: Effects of triggering on the oscilloscope display.

difficult aspects of learning to use the scope. Basically, the trigger circuit works by looking at some time-varying signal (called the trigger source) and generating

a trigger whenever that signal passes some preset level (which the user can adjust) with the proper slope (either positive or negative, as selected by the user). The trigger source can either be an external signal (EXT) provided by the user, the 60 Hz AC line voltage (LINE), or some other internal signal (INT). If the trigger source switch is set to INT then the trigger source will be one of the scope input channels (CH 1-4). One of these channels will be connected to your input signal but you have the ability to trigger on a different signal connected to one of the other channels. Edge triggering is the basic and most common type and is the only type discussed in this writeup. Consult the Tektronics website referenced above for details on other trigger types.

Common trigger modes include normal and auto. In normal mode the oscilloscope only sweeps if the input signal reaches the set trigger point; otherwise (on an analog oscilloscope) the screen is blank or (on a digital oscilloscope) frozen on the last acquired waveform. Normal mode can be disorienting since you may not see the signal at first if the level control is not adjusted correctly.

Auto mode causes the oscilloscope to sweep, even without a trigger. If no signal is present, a timer in the oscilloscope triggers the sweep. This ensures that the display will not disappear if the signal drops to small voltages. It is also the best mode to use if you are looking at many signals and do not want to bother setting the trigger each time. In practice, you will probably use both modes: normal mode because it is more versatile and auto mode because it requires less adjustment. Pulse measurements often require fine-tuning the triggering. Horizontal magnification is another useful feature for measuring pulses, since it allows you to see fine details of a fast pulse. The trigger position control is located in the horizontal control section of your oscilloscope, the Trigger **Level** knob. It actually represents "the horizontal position of the trigger in the waveform record." Horizontal trigger position control is only available on digital oscilloscopes. On your scope, the trigger appears as a small arrow pointing in on the right side of the screen. You adjust the trigger with your Trigger **Level** knob at the far right of the front panel. Push the Trig Menu button to view the options (they appear on the right side of the screen). **Set the Type to Edge, the Source to CH1, the Slope to Rising, the Mode to Auto, and the Coupling to DC.**

Other important knobs/dials:

Connect your signal cable to the **CH1** BNC fitting.

Push the **CH 1 Menu** button to set up additional options (see right side of screen): Select DC Coupling, BW Limit Off, Volts/Div Coarse (at least to start with), Probe 1X as mentioned above, and Invert Off.

Auto Set Button: A handy feature if you get lost in figuring out how to display a signal. This button reverts the display settings to factory default and you can start over.

On/Off Switch: Located on the top of the unit to the left.

SIGNAL PULSES

Signals such as steps and pulses that only occur once are called single-shot or transient signals. These are the signals you will observe during this course as part of setting up your detector systems. The step indicates a sudden change in voltage, like what you would see if you turned on a power switch. The pulse indicates what you would see if you turned a power switch on and then off again. It might represent one bit of information traveling through a computer circuit or it might be a glitch (a defect) in a circuit.

Voltage is the amount of electric potential (a kind of signal strength) between two points in a circuit. Usually one of these points is ground (zero volts) but not always - you may want to measure the voltage from the

maximum peak to the minimum peak of a waveform, referred to as the peak-to-peak voltage. The word amplitude commonly refers to the maximum voltage of a signal measured from ground or zero volts. In many applications, the details of a pulse's shape are important. Pulses can become distorted and cause a digital circuit to malfunction or create strange looking peaks in your spectra!

Standard pulse measurements are pulse width and pulse rise time. Rise time is the amount of time a pulse takes to go from the low to high voltage. By convention, the rise time is measured from 10% to 90% of the full voltage of the pulse. This eliminates any irregularities at the pulse's transition corners. This also explains why most oscilloscopes have 10% and 90% markings on their screen. Pulse width is the amount of time the pulse takes to go from low to high and back to low again. By convention, the pulse width is measured at 50% of full voltage.

OPERATIONS

Here's a brief description on how to get started:

1. Turn on the oscilloscope (button on top of unit to the left).
2. Connect your signal cable to either CH1 or CH2.
3. Put source in detector shelf (we're making the assumption that the detector system is already powered up).
4. Set up your Channel options. Push either CH1 or CH2 button (depending on which Channel has the signal cable connected to it). Go down the menu options and make the appropriate settings as discussed above.
5. Start adjusting your x and y scale to get the signal you want. You will probably also have to adjust the trigger level. You may play around with any of the buttons on the scope to see what they do. If you get lost, push the auto set button and you will be returned to the default settings.

Appendix 8: MCA (ScintiVision and GammaVision) Training

The NaI(Tl), Si, and HPGe detectors (gamma detectors) are controlled with computer based software provide by ORTEC®. This software is both a multichannel analyzer (MCA) emulator and a gamma-spectrum analysis program (ScintiVision® for NaI spectra, and GammaVision® for HPGe and Si spectra). You can familiarize yourself with the software using this guide and the handouts from the operating manual. Operating Manuals have been placed in Rms. 40 and 9 in B. 801 for reference. You may load on your laptop - but you have to bring your laptop to B. 801.

1. Start program: Click ScintiVision® or GammaVision® icon on computer desktop. The software opens to “detector” window. The MCA emulation continuously shows the currently acquiring spectra by whatever detector you have chosen to link the MCA with for your experiment. The full spectrum is displayed in the smaller window on the upper right side. The Expanded Spectrum window shows all or part of the full spectrum (see step 9 for changing the size of this window). Note: Spectrum acquisition continues if the computer is shut down, however the buffer is cleared each time software is turned off. One feature of this software is that since the computers in the laboratory are all connected to the LAN, each computer can see each detector. You choose your detector by selecting the appropriate name from the detector/buffer list (white window on the upper right portion of the screen). Your detector has the name of your computer, e.g. computer “ncss8” would control detector name ncss8 mcb #. **Be careful and never select a detector that does not include ncss in its name!**
2. Import spectra into software: Select white pull-down menu at upper right side of window and select “Buffer”. The buffer is a separate window on the computer screen and is maintained in the computer memory as a place where a spectrum can be moved for display and analysis either from the detector or from disk. The buffer is maintained independently from the detector operation so that you may collect spectra with the detector and simultaneously be analyzing a spectrum in the buffer. To import a spectrum from disk, select the **File** menu and then select “Recall”. Enter the information required. Sample spectra are located on the hard drive in the C:/User directory or on your personal z-drive.
3. Save a file: From either the buffer or detector window you may save a spectrum by selecting the **File** menu and “Save as”. Supply the required information (use .SPC format) and your spectrum will be saved accordingly. Alternatively, select the button that looks like a floppy disk on the upper left side of the menu above the window.

4. Move a spectrum from the detector window to the buffer window: Select the **Acquire** menu and select "Copy to buffer". You do not have to wait until the count is finished to move the current spectrum to the buffer.
5. Moving around the spectrum: The cursor may be moved using the space bar or by using the mouse. The cursor may also move from peak to peak by using the arrows in the Peak box (to the right of the spectrum).
6. Make an energy calibration (use minimum of two peaks which must span the energy region of your spectrum): Select the **Calibrate** menu and "Energy". Three windows open up: (1) an energy table listing the data points, (2) a graph of the calibration; and (3) the calibration window. Select the Windows icon on the calibration window. Select "Destroy" to get rid of a previous calibration. Select a peak (i.e. move the cursor to the peak) for which you know the energy. Enter the energy in the "E=" window of the calibration window. Select "Enter". Move to a second peak and repeat. The calibration is generated automatically by the software. When finished, close the calibration window. Save the calibration by selecting the **Calibrate** menu. Select "Save Calibration" and complete the required information.
7. Set Region of Interest (ROI): ROIs are a handy tool to determine net peak areas which are required to calculate activities and for monitoring the collection of counts. Around an area of interest in your spectrum (may be a peak or continuum region). Select the **ROI** menu and select "Mark". Now position the cursor using your mouse at the channel to start. Use the space bar to move the cursor to where you want to finish. This area will highlight. Again select the **ROI** menu and select "Off". ROIs are very useful because if you move the cursor to within the ROI and click, information about the region will appear in the gray bar below the spectrum window: Gross area and Net Area. If you have performed an energy calibration, the peak energy will also be displayed. Alternatively, you may see section 2.5.3 on p. 14 in the Maestro handout on a shortcut to do this.
8. More Peak Information: Move the cursor to the peak of interest (select the channel containing the maximum number of counts). Double click and a small window opens up with additional useful information like FWHM and counting error. If you are in Acquire mode, the window will tally the accumulation of counts too!
9. Change the Viewing Scale:
 - 9.1 The viewing scale can be changed from linear to log. Some spectrum features show up more clearly using log scale (Compton effects, background, small peaks in the vicinity of large peaks). For

- log scale, select the “Log” button which is located on the upper menu in the middle. Select the “A” button to view on a linear scale.
- 9.2 One may adjust the horizontal scale of the Expanded Spectrum Window by using the zoom in/out buttons (look like magnifying glasses and are located in the middle of the menu bar).
 - 9.3 Vertical Viewing Scale: May be adjusted using the up/down arrows on your keyboard.
10. Clear Spectrum: Select the button to the right of the “STOP” button on the top menu or select the **Acquire** menu and select “Clear”.
 11. Acquire Spectrum: Select the “Go” button. If you wish to specify a spectrum title and/or count time, use the **Acquire** menu: Select “Acquisition Settings” and select the options you desire such as “Acquisition Presets” for count time, “Sample Description” for a title. When you start acquiring your spectrum, boxes will open asking you to enter a title and a count time.
 12. Count Time Selection: It is important that you collect a sufficient number of counts in your peaks, preferably 4000 but certainly more than 1000. On the other hand, you don’t want to waste your time collecting for a longer time than is required. One way to monitor this is to create an ROI around your peaks of interest before you start your count. Then double click on your peak and a box will open which provides the net count information as it accumulates. Stop your collection once you have collected the required number of counts. In order to set up your ROIs, collect a quick spectrum that shows where the peaks are located. Then set up your ROIs, clear the spectrum and start a longer count. Stop it as described above. Consult your TA or Course Instructor if in doubt!
 13. Printing Spectra: You may print your spectra using Winplots. Operations Manuals are available for both in Rm. 40 and 9 (Look in the Maestro Manual, Chapter 6, p. 129).
For Winplots, basically:
 - 13.1 Open Winplots from the desktop.
 - 13.2 Go to file, recall spectrum in order to open your spectrum in Winplots.
 - 13.3 Go to file, print plot to print your spectrum for further analysis.

Ask the TAs or Course Instructor for further assistance.

Appendix 9: Generating Spectra for Lab Reports

1. Open Winplots and recall your spectrum: File\Recall\Your Spectrum
2. Once in Winplots, select File\Print. Select printing to the Microsoft Office Document Image Writer. Go into Properties and select the landscape option.
3. Print your spectrum. What will happen is that your spectrum will open up in the Microsoft Office Document Image Writer. In the Image Writer you can insert text boxes, highlight, etc...
4. Perform any additional editing as required by the Laboratory Report Guidelines.
5. Print your spectrum (the Image Writer will save the file to disk). Put the file in your folder at \\bnInt2\ncss.

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