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ABSTRACT

The experiments in this manual represent state-of-the-art techniques which should be within the budgetary constraints of a college physics or chemistry department. There are fourteen experiments divided into five modules. The modules are on X-ray fluorescence, charged particle detection, neutron activation analysis, X-ray attenuation, and accelerator experiments. Each module contains an introduction, a guide to the types of experiments contained, learning objectives, and a list of prerequisite skills. Each experiment contains objectives, an introduction, a list of equipment, data accumulation and analysis procedures, discussion of results, post-test questions, computer programs, optional work, and references. (Author/BB)
Advanced Experiments in Nuclear Science

Advanced Nuclear Physics
And Chemistry Experiments

Jerome L. Duggan, Floyd D. McDaniel,
and Jack G. Hehn

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And
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Denton, Texas 76203
INTRODUCTION

Many colleges and universities throughout the United States have recognized the importance of the study of nuclear science and radioisotopes in the undergraduate curriculum. The reasons for this realization are twofold: First, tomorrow's citizen is, without question, going to live in a world that is much more dependent on nuclear energy and technology than most of us realize at this time. Many of us think that the only practical and economical solution to the energy crisis is through nuclear energy at least until well after the year 2000. The ultimate energy needs of the United States will most likely be provided by a combination of numerous types of energy sources: nuclear fission and fusion, breeder reactors, solar and geothermal energy, as well as fossil fuels.

A second reason for studying radioisotopes lies in their application to certain analytical problems in chemistry, physics, biology, medicine and environmental science. As an example, neutron activation analysis is for many elements one of the most sensitive analytical techniques known. With the proper selection of pre and post chemical preparation techniques, sensitivities of the order of one nanogram may be obtained by this technique for many elements. X-ray fluorescence is another good example of a strong quantitative and qualitative technique that has recently been employed quite extensively for trace element analysis of environmental, geological, and biological samples. These techniques have been applied quite successfully not only to the areas of physics and chemistry but also to the areas of biology, geology, environmental science, and medicine. It is clear that today's science student needs a working knowledge of these "state of the art" techniques. This is the first of a series of manuals to be published in the
areas of physics and chemistry, biology, geology, and environmental science.

The experiments that are included in this manual for the most part represent "state of the art" techniques which should be within the budgetary constraints of a single physics or chemistry department. For smaller colleges or universities, it may perhaps be advantageous for a consortium of departments to share equipment costs. The experiments presented in the manual would probably be termed advanced; however many of these experiments have been performed successfully by undergraduates at various institutions inaugurating this text. A number of other fine texts and laboratory manuals have been written on what might be termed basic radiation measurements. For your convenience, we have listed a number of these in the general bibliography, Appendix VIII.

The laboratory manual contains a collection of five modules, each of which consists of one or more related or similar experiments. The modular approach allows one to present theoretical or introductory material in much greater detail for a collection of related experiments.

In general, each module will contain a one or two page introduction concerning the usefulness of the experiments and techniques to be studied and a guide to the different types of experiments to be encountered in the module; broad learning objectives of the module; and a prerequisites section which will contain a list of those necessary skills which every student should have before attempting the experiments in the module. The necessary prerequisites are given in the form of pre-test questions with answers to be found in the back of the module. Each module also contains a theoretical section which presents broad discussions of topics which are common to all of the experiments in the module. Following this introductory material are a number of similar experiments.
Each experiment contains specific objectives, an introduction, list of necessary and optional equipment, experimental data accumulation and analysis procedures, discussion of results, post-test questions to evaluate the students comprehension, computer programs available, optional work which may be done as an extension of the experiment, and additional references which may be used by the student to develop the experiment into a senior research project.

Following the collection of related experiments are the solutions to the pre-test and post-test questions for all of the experiments.

One should not necessarily expect a student to perform all of the experiments in a particular module but rather to do those experiments consistent with the goals of the institution's advanced laboratory course and the equipment available. The experiments are generally designed for a 3-4 hour laboratory period for student groups of 2 or 3. For some experiments, more than one laboratory period may be required.

The authors wish to acknowledge the valuable assistance received from the many consultants that have made this project possible. We also wish to thank the fifty or so reviewers of the earlier version of this manual for their valuable time and effort in evaluating these experiments.

While we have attempted to correct all errors in typing and procedures, some invariably will occur. We assume complete responsibility for any and all such errors and hope that future readers of this manual will inform us of them.

Jerome L. Duggan
Floyd D. McDaniel
Jack G. Hehn
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MODULE ONE

X-RAY FLUORESCENCE

A Module on Photon-Excited X-Ray Fluorescence

INTRODUCTION

X-ray fluorescence is a very powerful analytical technique which is used for the detection of trace impurities. Basically, the method involves bombarding a sample with photons from a radioactive source or other sources of photons while observing the characteristic X rays from the sample. The X rays produced in the sample are counted with a high-resolution X-ray detector and are characteristic of the elements present in the sample. With this method it is now possible to do quantitative analysis on a sample containing as many as twenty elements. The method has wide application in the fields of biology, chemistry, physics, geology, and environmental sciences and has also been used quite extensively for industrial problems and pollution analysis. At present, the detection limits are fairly well established at approximately 1.0 part per million. In addition to the sensitivities found in X-ray fluorescence studies, the method has the added advantage that it is a nondestructive type of analysis, i.e. the samples may be used again.

The module consists of six experiments which may be performed by the student. In the first three experiments the student will be introduced to the different detectors and techniques used in X-ray fluorescence studies. The advantages and disadvantages of the various detectors will be emphasized. The 4th and 5th experiments are more advanced and are designed for the student who has completed either Experiments 1 or 2 or has had some experience with Si(Li) or Ge(Li) detectors. Experiments 4 and 5 have environmental and geochemical applications. The 6th experiment is a tube-excited X-ray fluorescence experiment which represents the "state of the art" in photon-excited X-ray fluorescence. Sensitivities available with this technique are about a factor of 10 better than with source-excited methods but require a larger capital investment.

Following this introduction are the objectives for the module, a list of desirable prerequisites for the student to have before attempting this module, and a theoretical section which discusses some of the basic physical concepts and the nomenclature used in X-ray fluorescence studies.

OBJECTIVES

The objectives of the module are to familiarize the student with the detectors, equipment, and techniques used in photon-excited X-ray fluorescence. Commonly used X-ray detectors will
be employed to measure the energies of X rays produced in various samples. These energy spectra will be calibrated with X-ray sources of known energy. The calibrated spectrum will then be used to determine the energies of X rays from unknown sources, and hence, to identify the unknown elements present.

PREREQUISITES WITH PRE-TEST QUESTIONS AND PROBLEMS

1. Characteristics of Electromagnetic Radiation:
   a) wavelengths of different types,
   b) relationship between wavelength, frequency, and velocity, and
   c) relationship between energy and frequency of radiation, Planck's constant.

   1.1 What is the frequency of electromagnetic radiation that has a wavelength of one angstrom (10^-8 cm)?

   1.2 What is the energy in keV of a photon of this frequency?

2. Electron Volt (eV), keV, MeV:

   2.1 What is the definition of an electron volt?

   2.2 How is the eV related to the multiples keV, MeV, GeV?

3. Postulates of the Bohr Theory Of the Atom:

   3.1 State the postulates of the Bohr theory.

   3.2 An X-ray photon is emitted in an electronic transition between the K and L shells of an atom with a frequency of 8.45 x 10^-18 oscillations per second. What is the total energy difference between the two electronic orbits?

4. Coulomb Force:

   4.1 What is the magnitude and direction of the coulomb force between two electrons 5.0 cm apart?

   4.2 What is the nuclear coulomb barrier height in MeV for a 10 MeV alpha particle incident upon an atom of zinc of mass number A = 64?

5. Exclusion Principle:

   5.1 How does the exclusion principle apply to atoms?
6. Interaction of Electromagnetic Radiation with Matter:
   a) photoelectric effect
   b) Compton effect
   c) pair production

6.1 What is the maximum kinetic energy of an electron ejected from the surface of a metal by the photoelectric process if the work required to free the electron from the surface is 4 eV and ultraviolet light of frequency $10^{16}$ cycles/sec is used to illuminate the surface?

6.2 Show that for the Compton effect it is not possible to conserve both total relativistic energy and momentum if an essentially free electron absorbs all of the energy of an incident quantum.

7. Electron Capture and Internal Conversion:
   7.1 Explain the differences between electron capture and internal conversion.

   7.2 The ground state of $^{50}\text{V}$ decays by electron capture 70% of the time to the first excited state of $^{50}\text{Ti}$ at 1.55 MeV. How much energy is available for electron capture?

THEORY

The Bohr Atom:

X rays are electromagnetic radiation of wavelengths in the range from $10^{-6}$ to $10^{-9}$ cm. The origin of X rays can best be explained by referring to the semiclassical picture of the atom as proposed by Bohr. In this model the atomic electrons are assumed to move about the nucleus in well-defined, circular orbits under the influence of the coulomb attraction between the electron and the nucleus.

Figure 1 is a representation of the atomic nucleus and the first few electronic orbits. The orbits which are closest to the nucleus correspond to more negative energies. Electrons in these inner shells are more tightly bound to the nucleus than those in outer orbits.

Electromagnetic radiation is emitted if an electron, initially moving in a circular orbit of energy, $E_i$, discontinuously changes its motion so that it moves in an orbit of energy, $E_f$. The frequency of the emitted radiation, $v$, is equal to the quantity $(E_i - E_f)$ divided by Planck's constant, $h$. The radiation is classified as optical radiation or X radiation depending on its energy with no precise boundary separating the two. The optical radiation is lower in energy than the X radiation and includes the visible spectrum.
Electronic Energy Levels

The total energies of the electrons in the different orbits (i.e., $E_i$, $E_f$, etc.) may be found by solving Schrödinger's equation using the nuclear coulomb potential as the interaction potential between the negatively-charged electron and the positively-charged nucleus. For an atom of charge $+Ze$ with a single electron, this potential is just

$$V = \frac{-Ze^2}{r}$$

and one finds that the energy of this electron is just a function of the principal quantum number, $n$. The quantum number, $n$, may have the values $n=1, 2, 3$ and correspond to the major electronic shells K, L, M, ..., respectively. The solutions of Schrödinger's equation also identify two other quantum numbers labeled $l$, $m$, which correspond to the relative orbital angular momentum between the electron and the nucleus and its $z$-component. $l$ may have the values $l=0, 1, 2, ..., n-1$, and $m$ may have the values $m=-l, -l+1, -l+2, ..., 0, ..., l-2, +l-1, +l$. 

A diagram depicting the atomic nucleus and the first few electronic orbits.

Figure 1
Splitting of Energy Levels

In a one-electron atom the total energy of the electron does not depend on \( n \) or \( \ell \) since the potential is just the nuclear coulomb potential and is exactly proportional to \( 1/r \).

In a multi-electron atom the potential as seen by each electron is affected by the other electrons in the atom and is not exactly proportional to \( 1/r \). This is called the residual coulomb interaction. The total energy of the electron will depend on \( n \) as well as \( \ell \) and the result will be that the total energies of the electrons in the same shell will not be the same. For this reason, it is convenient to speak of each shell as being composed of a number of subshells, one for each value of \( \ell \). Each subshell has a capacity of \( 2\ell + 1 \) since the electrons may have \( z \)-components of orbital angular momentum \( m_\ell = -\ell \hbar, (-\ell+1)\hbar, \ldots, 0, (\ell-1)\hbar, \ell \hbar \). The exclusion principle requires that these quantum numbers be different for electrons in the same subshell.

If we further consider that electrons have an intrinsic spin angular momentum, \( S \), with \( z \)-components, \( M_S \), of \( +\hbar \) and \( -\hbar \), twice as many electrons may occupy each subshell and not violate the exclusion principle since each electron may have spin-"up" or "down." The total capacity of each subshell is therefore \( 2(2\ell+1) \). The energy ordering of a few of the subshells are given in Table I for different values of \( n \) and \( \ell \). The capacity of each subshell is given in the last column. The configuration of the atom is given by specifying the subshells occupied by the various electrons. Electronic configurations for a few atoms in the ground state are given below.

- **H**: \( 1s^1 \)
- **He**: \( 1s^2 \)
- **Li**: \( 1s^22s^1 \)
- **V**: \( 1s^22s^22p^63s^23p^64s^23d^3 \)

In the ground state the electrons must fill the subshells in such a way as to minimize the total energy of the atom and also not to exceed the capacity of \( 2(2\ell+1) \) electrons per subshell. The subshells are filled in order of increasing energy.

Figure 2 shows a typical energy level diagram and a few of the low energy subshells. There are an infinite number of discrete energy levels with \( E_{n\ell}=0 \) and a continuum of energy levels with \( E_{n\ell}>0 \).

Any further splitting of atomic energy levels is produced by the spin-orbit interaction between the magnetic moment of the electron spinning about its own axis and the magnetic field of the atom produced by the charged electron orbiting the nucleus.
### Table I

Energy Ordering of Electronic Subshells

<table>
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<tr>
<th>Quantum Number $n$</th>
<th>Subshell Designation</th>
<th>Capacity of Subshell</th>
</tr>
</thead>
<tbody>
<tr>
<td>4</td>
<td>3</td>
<td>4f</td>
</tr>
<tr>
<td>6</td>
<td>0</td>
<td>6s</td>
</tr>
<tr>
<td>5</td>
<td>1</td>
<td>5p</td>
</tr>
<tr>
<td>4</td>
<td>2</td>
<td>4d</td>
</tr>
<tr>
<td>5</td>
<td>0</td>
<td>5s</td>
</tr>
<tr>
<td>4</td>
<td>1</td>
<td>4p</td>
</tr>
<tr>
<td>3</td>
<td>2</td>
<td>3d</td>
</tr>
<tr>
<td>4</td>
<td>0</td>
<td>4s</td>
</tr>
<tr>
<td>3</td>
<td>1</td>
<td>3p</td>
</tr>
<tr>
<td>3</td>
<td>0</td>
<td>3s</td>
</tr>
<tr>
<td>2</td>
<td>1</td>
<td>2p</td>
</tr>
<tr>
<td>2</td>
<td>0</td>
<td>2s</td>
</tr>
<tr>
<td>1</td>
<td>0</td>
<td>1s</td>
</tr>
</tbody>
</table>

**Figure 2**

Atomic energy levels showing a few low energy subshells.
Coupling of Angular Momentum

For atoms with small and intermediate atomic number, $Z$, the residual coulomb interaction is much larger than the spin-orbit interaction and the individual spin angular momenta of the optically active electrons couple to form a total spin angular momentum for the atom. For an atom with $i$ optically active electrons (i.e., electrons in unfilled shells),

\[ \mathbf{S} = \mathbf{S}_1 + \mathbf{S}_2 + \ldots + \mathbf{S}_i \]

$\mathbf{S}$ has the constant magnitude $S = \sqrt{s(s+1)}\hbar$ and $z$-component $S_z = m_s\hbar$. The individual orbital angular momentum couple in the same manner to form a total orbital angular momentum for the atom:

\[ \mathbf{L} = \mathbf{L}_1 + \mathbf{L}_2 + \ldots + \mathbf{L}_i \]

which has the constant magnitude $L = \sqrt{l(l+1)}\hbar$ and $z$-component $L_z = m_l\hbar$.

The smaller spin-orbit interaction then couples the total spin angular momentum, $\mathbf{S}$, and the total orbital angular momentum, $\mathbf{L}$, to form the total angular momentum,

\[ \mathbf{J} = \mathbf{L} + \mathbf{S} \]

which again has the constant value $J = \sqrt{j(j+1)}\hbar$ and $z$-component $J_z = m_j\hbar$. This is called LS coupling or Russell-Saunders coupling.

For atoms with larger atomic number, $Z$, the spin-orbit interaction is larger than the residual coulomb interaction. The spin-orbit interaction first couples the individual spin and orbital angular momentum of each electron to form a total angular momentum for each electron, i.e., $\mathbf{J}_1 = \mathbf{L}_1 + \mathbf{S}_1$. The weaker coulomb interactions then couple the total angular momentum for each electron, $\mathbf{J}_1$, to form a total angular momentum for the atom,

\[ \mathbf{J} = \mathbf{J}_1 + \mathbf{J}_2 + \ldots + \mathbf{J}_i \]

This type of coupling is called JJ coupling.

The energy levels produced by either type of coupling correspond to total angular momenta, $\mathbf{J}$, which has the constant magnitude $J = \sqrt{j(j+1)}\hbar$ and $z$-component $J_z = m_j\hbar$. 
The atomic energy levels are thus specified by the quantum numbers $n$, $\ell$, $j$.

Interaction of Electromagnetic Radiation with Matter

Electromagnetic radiation may interact with the atomic electrons by three processes: photoelectric effect, Compton effect, and pair production. In the photoelectric effect the energy of the incident quantum is completely absorbed by the atom and the bound electron is ejected. For a Compton encounter the quantum scatters from an essentially free electron and goes off suffering only a reduction in energy. For quanta with energies greater than the rest mass energy of a pair of electrons, the process of pair production is possible. Here, the quantum of electromagnetic radiation completely disappears with the production of a pair of electrons.

Excited States of Atoms

For an atom in an excited state, one or more electrons are excited to a higher energy level (more positive in energy). Fortunately, in most cases the number of electrons which make the transitions is small. If an atom is excited by the absorption of electromagnetic radiation, at most only one quantum is absorbed by any particular atom and the quantum tends to interact with only one or two electrons. The configuration of an atom in an excited state differs from an atom in its ground state in that one or at most a few electrons are in higher energy subshells and there are corresponding vacancies in the subshells from which they came.

The possible excitation of an atom may be divided into two primary types. The first type corresponds to excitations in which an electron in one of the higher energy subshells which were occupied in the ground state of the atom is excited. The electron may be excited into a higher energy bound state which corresponds to a negative total energy or into a positive energy continuum state. If the excitation of this electron is completely out of the atom into a continuum state, the atom is said to be ionized. The second type of excitation corresponds to an electron in one of the lower energy subshells being promoted to a higher energy unoccupied subshell or to a continuum state. The exclusion principle requires that the electron be excited to an unfilled subshell rather than one that is already filled. These two types of excitations are represented in Figure 2.

X-Ray Emission

An excitation of the first type requires only a few electron volts (eV) of energy while an excitation of the second type may require more than a 1000 eV. After an excitation of either type the atom will eventually return to its ground state by producing
quanta of radiation. The very high energy quanta that form the characteristic X-ray spectrum of the atom are emitted when an excitation of the second type has taken place. The much lower energy quanta emitted when an atom returns to its ground state after an excitation of the first type form the optical spectrum of the atom. The atom de-excites in both cases as electrons in the higher energy levels fall into the holes left by the excited electrons.

The major difference between X-ray and optical spectra is that the energies of X rays increase uniformly from element to element as the atomic number, Z, increases and has no abrupt changes in energy from one element to the next as in optical spectra. The reason being that the energies of the characteristic X rays depends on the binding energies of the electrons in the inner shells. With increasing atomic number, Z, these binding energies simply increase uniformly due to the increased nuclear charge, and are not affected by the periodic changes in the number of electrons in the outer shell. The atomic energy levels involved in the emission of X rays are shown in Figure 3 for four of the innermost major shells. The energy levels are specified by the quantum numbers \( n, \ell, j \) which are given adjacent to the levels in Figure 3. The various possibilities for the K and L X-ray lines are also shown.

If the photoelectric effect occurs and removes a K electron from the atom, the most probable event is an L electron will fall into the K shell vacancy producing the characteristic K radiation from that element in the sample. For example, the radiation produced when a LIII electron falls into a K vacancy is called the K\(_1\) characteristic X ray. If an MIII electron falls into the K shell vacancy a K\(_{2,3}\) X-ray is produced. For light elements (i.e. below Tin) the most prominent X ray from an element are the K\(_1\) and the K\(_{3}\) lines.

Not all transitions are possible in Figure 3. There is a set of selection rules governing the changes in quantum numbers for the allowed transitions. The selection rules are:

\[
\Delta n = \pm 1,
\Delta \ell = 0, \pm 1
\]

Some of the possible transitions which obey these rules are shown in Figure 3 for the K and L X rays. The X-ray line spectrum of the atom consists of these allowed transitions plus a few which are observed to be emitted very infrequently in violation of these rules.
ATOMIC ENERGY LEVELS INVOLVED IN THE EMISSION OF X-RAYS

Figure 3
Bremsstrahlen

In addition to the X-ray line spectrum, one may also observe a continuum spectrum which is often called bremsstrahlen. This is a word of German origin which means braking radiation or deceleration radiation. Bremsstrahlen is produced by electrons, which have been ejected from the atom, that have suffered Coulomb interaction scattering in close collisions with the nuclei of other atoms in the target.
Experiment 1

Basic Source-Excited X-Ray Fluorescence with a Lithium-Drifted Silicon Detector

Objective

To study the basic properties of a lithium-drifted silicon detector for X-ray measurements; to study the techniques of preparing samples for fluorescing; to construct a calibration curve with known X-ray and gamma-ray lines and to use the calibration curve to determine the elemental composition of the unknown samples; to measure the resolution of a Ge(Li) detector; to determine the energies of X rays from unknown radioactive sources.

References


Introduction

In this experiment we are going to use a Si(Li) detector to measure the X rays as low in energy as 2 keV and up to about 30 keV. Normally these detectors show resolution capabilities of 200 eV or better. Figure 3, for example, shows a pulse height spectrum of Mn with one of these detectors. From an analytic point of view one of the most difficult problems in X-ray spectroscopy (non-dispersive) is in trying to resolve between adjacent elements in the periodic table. For example, let us assume that an unknown sample contains zinc and copper. The strong X-ray line that we
would see from Zn is the Kα line at 8.638 keV. The Kα line from copper is 8.047 keV. (These energies are listed in Appendix V of this manual: X-Ray Critical Absorption and Emission Energies.) The problem is resolving these two lines which are 591 eV apart in energy. This can quite easily be done with a 200 eV resolution detector.

If the student makes a careful calibration curve in accordance with the instructions in this report, he will easily be able to identify the unknowns that are provided in the latter part of the experiment.

Equipment

1. Lithium-Drifted Silicon X-Ray Detector and Pre-amplifier (resolution ~200 eV)
2. Detector Bias Supply
3. Low Noise Spectroscopy Amplifier
4. Multichannel Analyzer (~500 channels)
5. Excitation Source: 25 mCi of ¹⁷⁶Yb (The source must be specially prepared for fluorescence applications. See, for example, New England Nuclear Corporation Radioactive Source Catalog, 36 Pleasant Street, Watertown, Massachusetts. Note: sources of this activity require state licensing.)
6. Oscilloscope (optional)
7. Various Materials to be Fluoresced (for example: titanium foil, copper foil, arsenic powder, and yttrium metal)
8. Nuclear Pulse Generator (optional)

Procedure

1. Sample Preparation for Calibration

The materials to be used for samples can be standard reagent grade chemicals. Usually these chemicals are 95-plus percent pure. Metallic foils can be mounted directly above the fluorescing apparatus or in an inexpensive sample holder. Powder samples are usually placed in these plastic sample holders. These holders or cups are designed so that the part of the holder that faces the X-ray detector is a 1.25 × 10⁻³ cm Mylar film. The cups are plastic cylinders that have Mylar stretched over the bottom of the cylinder.

The Mylar offers very little attenuation either to the exciting photon coming into the sample or the fluorescence X rays coming out of the medium. The plastic cups and the Mylar are nearly free of trace
materials and hence, quite good for trace analysis work. These sample cups can be obtained from any of the companies that provide supplies for X-ray analysis studies. One such company is: Somas Laboratories Inc., 54 East 11th Street, New York, NY 10003.

Many manufacturers supply cups that can handle either solid or liquid samples. In general sample preparation for calibration is quite simple and offers no real problem for the student.

The samples to be used in this experiment can be prepared by the students before the laboratory period begins.

2. Electronics.

Set up the electronics as shown in Figure 1. Actually nuclear modular electronics is quite easy to "hook up." Inputs and outputs are usually clearly labeled and the lengths of connecting cables are in general not important. Make sure the instructor approves the set up before turning on the high voltage power supply. CAUTION On some Si(Li) detectors the high voltage must be slowly turned to the recommended value. A wise practice is to take about 1 minute to go from zero up to the recommended voltage.

![Figure 1](image)

**Figure 1**

Electronics for Source Excited X-ray fluorescence.

Usually the source and collimator are purchased from the manufacturer as a single unit. Ideally, this unit would fit snugly over the top of the detector shown in Figure 1. Since the sources used for X-ray fluorescence analysis are of the order of many mCi's, some precaution should be used in handling the sources. The recommended procedure is usually included by the manufacturer in the instruction manual. Care must also be taken to protect the thin
berrylium window that allows the X rays to pass into the sensitive region of the detector.

These windows are of the order of $2.5 \times 10^{-3}$ cm or thinner; hence, they will definitely break if they are touched, even lightly.

3. Calibration

a. Place the highest atomic number sample to be used in the sample position (Figure 1)—in our case, this is yttrium, $Z = 39$. The sample should physically be about 2.0 cm or closer from the face of the detector. Adjust the gain of the amplifier so that the 14.933 keV $K_{\alpha}$ line from yttrium appears about midway in the multichannel analyzer. This roughly calibrates the system to 30 keV full scale.

b. Accumulate an yttrium spectrum in the multichannel analyzer for a time period long enough to have ~1000 peak counts in the $K_{\alpha}$ line. This should take about 3 minutes. Readout the multichannel analyzer on the teletype and record the channel position of the $K_{\alpha}$ and $K_{\beta}$ lines. Note, a computer program, written in BASIC LANGUAGE (GAUSS-6) is included at the end of this manual which will find the centroid of a GAUSSIAN peak to an accuracy of 0.1 channels. Actually for the purposes of this experiment, a visual estimate of the centroid from the multichannel will usually suffice.

c. Accumulate spectra of arsenic, copper, and titanium, and determine the centroids of the $K_{\alpha}$ and $K_{\beta}$ lines in the same manner as (b).

d. Plot a curve of energy vs channel number to establish the calibration curve. Table I shows typical calibration data that were obtained by a group of undergraduate environmental students from Stockton State College, Pomona, New Jersey. Figure 2 shows the corresponding calibration curve for this data. In Table I the column labeled "Peak Channel from Readout" is the rough estimate of where the peak is from the teletype output. The column labeled "Gaussian Fit" was obtained from the computer program GAUSS-6.

e. Obtain the unknowns from the instructor, accumulate their spectra, find the centroids of the lines, assign energies from the calibration curve; and, hence, determine what elements are present in the unknowns. Figures 4 and 5 show typical compounds that are given as unknowns. Remember for $K$ lines the $K_{\alpha}$ and $K_{\beta}$ energies must be correct, or at least within 100 eV of the accepted values. The same is true for $L$ lines, which are shown in Figure 6. $L_{\alpha}$, $L_{\beta}$, and $L_{\gamma}$ must have the proper energies.
### Table I

<table>
<thead>
<tr>
<th>Sample</th>
<th>Line</th>
<th>Energy (keV) Theory</th>
<th>Peak Channel from Readout</th>
<th>Peak Channel Gaussian Fit</th>
</tr>
</thead>
<tbody>
<tr>
<td>Titanium</td>
<td>K_α</td>
<td>4.508</td>
<td>75</td>
<td>75.2</td>
</tr>
<tr>
<td>Titanium</td>
<td>K_β</td>
<td>4.931</td>
<td>83</td>
<td>83.17</td>
</tr>
<tr>
<td>Copper</td>
<td>K_α</td>
<td>8.041</td>
<td>143</td>
<td>143.29</td>
</tr>
<tr>
<td>Copper</td>
<td>K_β</td>
<td>8.907</td>
<td>160</td>
<td>160.0</td>
</tr>
<tr>
<td>Arsenic</td>
<td>K_α</td>
<td>10.532</td>
<td>192</td>
<td>191.5</td>
</tr>
<tr>
<td>Arsenic</td>
<td>K_β</td>
<td>11.729</td>
<td>214</td>
<td>214.63</td>
</tr>
<tr>
<td>Yttrium</td>
<td>K_α</td>
<td>14.933</td>
<td>277</td>
<td>277.41</td>
</tr>
<tr>
<td>Yttrium</td>
<td>K_β</td>
<td>16.754</td>
<td>312</td>
<td>312.4</td>
</tr>
</tbody>
</table>

#### Figure 2

Calibration curve for Si(Li) detector.
In general, if one is careful in making the calibration curve, it is difficult to make a mistake on what elements are present in the sample.

Data Analysis and Discussion

This method of qualitative analysis is a very strong analytic technique. The student is urged to read References 1, 2, 4, 5, and 6. Most of these are short articles which are written in a manner such that an advanced undergraduate student will understand most of the content of the articles.

After reading the articles and having done this laboratory experiment, the student will have a better appreciation for the wide applicability of this technique.

In order to do quantitative analysis on an unknown sample, the problem is a bit more difficult. Calculations can be made that will give the number of X rays that one would expect to see from a given source target configuration; but in general, they are quite involved.
Figure 4

K X-ray spectra from typical chemical unknowns given to students.
Figure 5

K X-ray spectra of mixed unknown samples.
The first problem is calculating how many exciting photons get into the sample and how deep; next, the question is how many characteristic X rays get out of the sample and into the detector. There are at least six parameters that one must consider for a single element target.

Resolution of the Detector

Shown in Figure 2 is the slope of the calibration curve. This was obtained by determining the quantity \( \frac{dE}{dc} \) where \( dE \) is the difference in energy between the Y K\(_\beta\) and the Ti K\(_\alpha\), \( dc \) is the corresponding channel difference. The resolution of the detector is the channel spread across the peak multiplied by the slope of the calibration curve. Detector resolution is usually given in terms of the Mn K\(_\alpha\) line. For example, Figure 3 shows a pulse height spectrum that was obtained for Mn with a system that had a calibration slope of 30.18 eV/channel. The channel spread across the peak (K\(_\alpha\)) in Figure 3 is about 5.5 channels. Note, it takes 6 plotted channels to give a 5-channel spread, or full width at half maximum (FWHM) as it is sometimes called.

Hence, for Figure 3:

\[
\text{Resolution} = (5.5 \text{ channels})(30.18 \text{ eV/channel})
\]

\[
= 166 \text{ eV}
\]

Determine the resolution of your detector by the method outlined above.

Determination of an Unknown

At this point the student is ready for the unknowns. In general, the instructor will have these samples prepared and ready to give to the students. These unknowns can be single elements, chemical compounds, or solutions. The student should remember that both K and L lines can be excited from the sample. If the unknown contains K lines, a spectrum similar to Figure 3 will be obtained. The K\(_\alpha\) peak has approximately seven times as many counts as the K\(_\beta\). For L lines the groups usually observed are the L\(_\alpha\), L\(_\beta\), and L\(_\gamma\). For example, Figure 6 shows typical L lines from a gold foil.

An easier method to do quantitative analysis with fluorescence is by the comparative method. In using this method, all samples must be thin. This means that X rays made at the deepest portion of the sample must not be attenuated appreciably in going through the sample to the detector.

For example, let us assume that we have a dried water residue that contains iron. We fluoresce a thin sample of the residue (~0.008 cm thick) and obtain the iron peaks. Next a thin (known
weight) sample of iron sulfate is studied. By comparing the number of counts under the Fe-Kα peaks for these two samples, a good estimate of the quantity of iron in the water residue can be determined by using the ratio method. Other environmental samples can be studied in the same way.

Post-Test:

1.1 Why do the energies of X rays increase rather uniformly with atomic number Z, while the energies of optical lines are so non-uniform even for adjacent elements in the periodic table?

1.2 How many disintegrations per second are there from a source of 0.1 mCi activity?

1.3 What is the function of the collimator in Figure 1?
1.4 Roughly estimate the number of counts in a gaussian peak which is 6 channels wide (FWHM) and contains 3000 counts in the peak channel.

1.5 Obtain the slope of the calibration curve in Figure 2 using the data given in Table I for a) the Titanium K_α and Arsenic K_β lines, and b) the Copper K_α and the Yttrium K_α lines.

1.6 If a radioactive source of intensity I_0 has a half-life of 128 days, what will be the intensity in 640 days? How many half-lives is this?

1.7 If the spectroscopy amplifier shown in Figure 1 is a linear amplifier, does that mean that the calibration curve shown in Figure 2 will also be linear?

**Computer Programs**

GAUSS-6 finds centroids and FWHM of pulse height spectra. LINEAR-6 does linear least squares fit to calibration data. This program can be used to very accurately establish the calibration curve. Students have used this program to identify unknowns to ±30 eV.

**Optional Work**

Obtain radioactive sources from the instructor that have X rays in their decay scheme. Determine the energies of the X rays from these sources. Any radioactive source (~1 μCi) that has either electron capture or internal conversion in its decay scheme will show X rays from the daughter atom. The student is urged to review these two simple nuclear processes. For this experiment the ¹⁰⁹Cd source is removed and the source card containing the source is placed about 1 cm away from the detector.

Figure 7 shows decay schemes for ⁵⁵Fe, ⁵⁷Co, ⁵⁴Mn, and ⁶⁵Zn. These radioactive sources can be purchased from many of the vendors that are listed in the appendix of this manual. It should be noted that when ordering sources for X-ray work, it is necessary to indicate to the manufacturer that the sources should have a thin Mylar window for this application. Table II gives a tabulation of common X-ray sources and the energies that are emitted by the sources.

The efficiency of the Si(Li) detector can be determined by purchasing standard sources from the source manufacturers. This point will become more important to the student who is trying to do both qualitative and quantitative analysis on samples. Most of the manufacturers will supply standard activity sources for X-ray work. The problem is then to measure the efficiency of the detector at a fixed distance from the face of the detector. Figure 8 shows...
Figure 7
Decay schemes for $^{57}$Co, $^{54}$Mn, $^{55}$Fe, and $^{65}$Zn.

Table II
Common Sources for X-ray Calibration

<table>
<thead>
<tr>
<th>Nuclide</th>
<th>Daughter</th>
<th>Half-Life</th>
<th>Radiation</th>
<th>Photon Energy (keV)</th>
</tr>
</thead>
<tbody>
<tr>
<td>$^{57}$Co</td>
<td>$^{57}$Fe</td>
<td>271.6 d</td>
<td>$K_\alpha, K_\beta$ X rays</td>
<td>6.40, 7.059</td>
</tr>
<tr>
<td>$^{54}$Mn</td>
<td>$^{54}$Cr</td>
<td>312.6 d</td>
<td>$K_\alpha, K_\beta$ X rays</td>
<td>5.411, 5.947</td>
</tr>
<tr>
<td>$^{88}$Y</td>
<td>$^{88}$Sr</td>
<td>106.6 d</td>
<td>$K_\alpha, K_\beta$ X rays</td>
<td>14.933, 16.754</td>
</tr>
<tr>
<td>$^{241}$Am</td>
<td>$^{237}$Np</td>
<td>$1.58 \times 10^5$ d</td>
<td>$L_\alpha, L_\beta$ X rays</td>
<td>13.9, 17.8, 20.8</td>
</tr>
<tr>
<td>$^{241}$Am</td>
<td>$^{237}$Np</td>
<td></td>
<td>$M$ X rays</td>
<td>3.30</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>$\gamma$ ray</td>
<td>26.4</td>
</tr>
<tr>
<td>$^{203}$Hg</td>
<td>$^{203}$Tl</td>
<td>46.59 d</td>
<td>$K_\alpha, K_\beta$ X rays</td>
<td>70.821, 69.894</td>
</tr>
<tr>
<td>$^{58}$Fe</td>
<td>$^{58}$Mn</td>
<td>2.7 y</td>
<td>$K_\alpha, K_\beta$ X rays</td>
<td>5.895, 6.492</td>
</tr>
<tr>
<td>$^{65}$Zn</td>
<td>$^{65}$Cu</td>
<td>243.7 d</td>
<td>$K_\alpha, K_\beta$ X rays</td>
<td>8.041, 8.907</td>
</tr>
<tr>
<td>$^{51}$Cr</td>
<td>$^{51}$V</td>
<td>27.8 d</td>
<td>$K_\alpha, K_\beta$ X rays</td>
<td>4.949, 5.427</td>
</tr>
</tbody>
</table>
typical efficiencies of a Si(Li) detector with various thickness entrance windows. This experiment can easily be done if the standard sources are available. Figure 9 shows a typical spectrum that was obtained from an $^{241}$Am source. The intensities of the lines in Figure 9 are shown in Table III. The platinum peaks shown in Figure 9 are from fluorescence of the source backing.

![Figure 8](image)

Spectral response of a Si(Li) detector.

Table III

<table>
<thead>
<tr>
<th>Photon Energy (keV)</th>
<th>Relative Intensity %</th>
</tr>
</thead>
<tbody>
<tr>
<td>11.89 Np L$_{\alpha}$</td>
<td>2.2</td>
</tr>
<tr>
<td>13.90 Np L$_{\alpha}$</td>
<td>37.5</td>
</tr>
<tr>
<td>17.80 Np L$_{\beta}$</td>
<td>56.3</td>
</tr>
<tr>
<td>20.80 Np L$_{\gamma}$</td>
<td>13.8</td>
</tr>
<tr>
<td>26.35 Am- $\gamma$</td>
<td>7.0</td>
</tr>
<tr>
<td>59.50 Am- $\gamma$</td>
<td>100.0</td>
</tr>
</tbody>
</table>
Additional References

For students that wish to develop this technique into a student research type problem, the following references will be helpful.


Experiment 2
X-Ray Fluorescence with a Lithium Drifted Germanium Detector (Source Excited)

Objective

To study the basic properties of a lithium-drifted germanium, Ge(Li), detector; to construct a calibration curve with known X-ray and gamma-ray lines from radioactive sources and to study source-excited X-ray fluorescence in the energy range from 15 to 100 keV; to determine the elemental composition in unknown samples.

References


Introduction

In this experiment we will use a Ge(Li) detector to study source-excited X rays. These detectors are perhaps more versatile than the Si(Li) detectors because they have a broader energy range. In general, they can be used to look at X rays in the range from 5 keV to 130 keV. From the Critical Absorption and Emission X-Ray Chart in the appendix of this manual, it appears that with these detectors it is possible to study K lines of elements from Z = 23 (Vanadium) all the way through the end of the periodic chart. Ultra-thin-window devices have also been manufactured that will measure energies down to 2 keV. The resolutions of these Ge(Li) detectors are usually not as good as one will find for Si(Li) devices. Typical values are 300 eV at an energy of 6 keV.
Nevertheless, they represent the "state of the art" for looking at K lines for intermediate to heavy elements. Fortunately, nature is with us here in that as the atomic number of the sample increases the K X-ray energy difference between adjacent elements also increases; and, hence, 300 eV resolution is adequate for most measurements. In any case, once the student has performed this experiment, he will certainly appreciate the usefulness of these detectors. In a later experiment we will discuss the use of these thin-window Ge(Li) X-ray detectors in the solution to pollution problems.

**Equipment.**

1. Lithium-Drifted Germanium, Ge(Li), Beryllium Window X-Ray Detector (resolution 400 eV or better at 6 keV)
2. Detector Bias Supply
3. Low Noise Spectroscopy Amplifier
4. Multichannel Analyzer (≈500 channels)
5. Radioactive Sources for Calibration as follows:
   a) 10 μCi $^{57}$Co
   b) 1 μCi $^{241}$Am
   c) 5 μCi $^{137}$Cs
   d) 2 μCi $^{109}$Cd
6. Radioactive Source for Exciting the Samples 10 mCi $^{57}$Co
7. Oscilloscope
8. Various Materials to be Fluoresced (for example: silver, tin, iodine, tantalium, gold, mercury, lead)
9. Nuclear Pulse Generator (optional)

**Procedure**

1. Electronics

   Set up the electronics as shown in Figure 1. Note, care should be used in adjusting the bias voltage to its recommended value. The collimator shown in Figure 1 is a lead sleeve that fits over the end cap of the detector. The sleeve has a solid end on it which faces the sample. A hole (≈1.25 cm) is drilled in the solid end which allows the emitted X rays to pass into the detector. Usually the schematic drawings that come with the excitation source (10 mCi $^{57}$Co) will clearly indicate how the source can be adapted to the detector. The radioactive sources are usually deposited in ring
geometry directly on the collimator. The samples to be studied should be placed about 2 cm from the excitation source.

Figure 1
Electronics for X-ray fluorescence with a Ge(Li) detector.

2. Calibration

In this experiment we are going to study X rays in the energy range from 10 to 85 keV; and, hence, the multichannel analyzer must be calibrated in this range. This calibration will be made with X-ray and gamma-ray lines from radioactive sources. The sources used and their characteristic energies are shown in Table I.

Table I
Radioactive Sources Used for Energy Calibration Ge(Li)

<table>
<thead>
<tr>
<th>Source</th>
<th>Half-Life</th>
<th>Photon Energies (KeV)</th>
</tr>
</thead>
<tbody>
<tr>
<td>$^{57}$Co</td>
<td>270 d</td>
<td>14.37, 121.94, 136.31</td>
</tr>
<tr>
<td>$^{241}$Am</td>
<td>458 y</td>
<td>26.36, 59.57</td>
</tr>
<tr>
<td>$^{137}$Cs</td>
<td>30.0 y</td>
<td>32.191, 36.376</td>
</tr>
<tr>
<td>$^{109}$Cd</td>
<td>453 d</td>
<td>87.7, 22.162, 24.942</td>
</tr>
</tbody>
</table>
Figures 2 and 3 show the characteristic decay modes of these sources.

**Figure 2**
Decay schemes for $^{57}$Co, $^{109}$Cd, and $^{137}$Cs.

**Figure 3**
Decay scheme for $^{241}$Am.
Figure 4 shows a high resolution spectrum of $^{57}\text{Co}$. In this experiment we are not interested in the Fe $K_a$ and $K_b$ lines since they are below our calibration range. They are shown only to indicate that the electron capture process yields characteristic X rays from the daughter atom which in this case is iron. $^{241}\text{Am}$ is a little more complicated. Figure 5 shows a spectrum of $^{241}\text{Am}$ which indicates some of this complexity in the lower energy range of the detector. In the spectrum the L X rays come from the strong internal conversion coefficients for the levels in $^{237}\text{Np}$. The Pt L-lines originate from fluorescence of the Pt blank that served as a backing for electrodeposition of the source. Figure 6 shows a composite spectrum of $^{57}\text{Co}$, $^{65}\text{Zn}$, and $^{85}\text{Sr}$. This figure illustrates the resolution capabilities of a Ge(Li) detector at low energies.

With the lines indicated in Table I, make a plot of energy vs channel number. From the plot, determine the slope of the
Figure 5
Pulse height spectrum of $^{241}$Am.

Figure 6
Composite spectrum of $^{57}$Co, $^{65}$Zn, and $^{85}$Sr.
calibration curve. Figure 7 shows a similar plot that was made for a Ge(Li) detector used in this manner.

![Calibration curve for Ge(Li) showing data points.](image)

**Figure 7**
Calibration curve for Ge(Li) showing data points.

3. **${}^{57}\text{Co}$ Excitation of Various Elements**

Replace the $^{57}\text{Co}$ source exactly as shown in Figure 1. Place a piece of silver in position to be irradiated with the $^{57}\text{Co}$ gammas. Accumulate a spectrum for a period of time long enough to get at least 1000 counts in the Ag K$_\beta$ group. Readout the spectrum and replace the Ag metal with a piece of tin. Continue running samples until you have run all of those that the laboratory instructor has provided.

4. **${}^{57}\text{Co}$ Excitation of Unknowns**

Obtain the unknown samples from the laboratory instructor, fluoresce them, and obtain their spectra as in 3. Figure 8 shows a composite of Ag, Cd, In, and Sn. These are shown to illustrate that adjacent elements near silver can easily be resolved. In a real situation, the K$_\beta$ groups can be subtracted out from under
the $K_{\alpha}$ groups. This is easy to do since the $K_{\alpha}/K_{\beta}$ ratios are well known and the position of the $K_{\beta}$ group is also accurately known. Figures 9, 10, and 11 show $K$ lines from three popular samples that are frequently studied. Note the separation of the $K_{\alpha 2}$ and $K_{\alpha 1}$ lines for heavy elements. For light elements the $K_{\alpha 1}$ and $K_{\alpha 2}$ groups usually appear as one group.

Data Reduction

Exercise (a)

Measure the resolution of the following lines from the calibration sources used in the CALIBRATION (B) 14.37, 22.162, 32.191, 59.57, and 122 keV. See Experiment 1 for the details of this procedure. Fill in the data Table II.

Exercise (b)

Plot a curve of energy in keV versus resolution from the data in Table II.

<table>
<thead>
<tr>
<th>Line</th>
<th>Energy (keV)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Ag $K_{\alpha}$</td>
<td>22.104</td>
</tr>
<tr>
<td>Ag $K_{\beta}$</td>
<td>24.987</td>
</tr>
<tr>
<td>Cd $K_{\alpha}$</td>
<td>23.109</td>
</tr>
<tr>
<td>Cd $K_{\beta}$</td>
<td>26.143</td>
</tr>
<tr>
<td>In $K_{\alpha}$</td>
<td>24.139</td>
</tr>
<tr>
<td>In $K_{\beta}$</td>
<td>27.382</td>
</tr>
<tr>
<td>Sn $K_{\alpha}$</td>
<td>25.193</td>
</tr>
<tr>
<td>Sn $K_{\beta}$</td>
<td>28.601</td>
</tr>
</tbody>
</table>
Figure 9
K X-ray spectrum of gold.

<table>
<thead>
<tr>
<th>Line</th>
<th>Energy (KeV)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Au-K\textsubscript{a1}</td>
<td>66.980</td>
</tr>
<tr>
<td>Au-K\textsubscript{a1}</td>
<td>68.794</td>
</tr>
<tr>
<td>Au-K\textsubscript{a1}</td>
<td>77.968</td>
</tr>
<tr>
<td>Au-K\textsubscript{a1}</td>
<td>80.165</td>
</tr>
</tbody>
</table>

Figure 10
K X-ray spectrum of tungsten.

<table>
<thead>
<tr>
<th>Line</th>
<th>Energy (keV)</th>
</tr>
</thead>
<tbody>
<tr>
<td>W K\textsubscript{a1}</td>
<td>57.973</td>
</tr>
<tr>
<td>W K\textsubscript{a1}</td>
<td>59.318</td>
</tr>
<tr>
<td>W K\textsubscript{a1}</td>
<td>67.233</td>
</tr>
<tr>
<td>W K\textsubscript{a1}</td>
<td>89.696</td>
</tr>
</tbody>
</table>
### Table II

<table>
<thead>
<tr>
<th>Photon Energy (keV)</th>
<th>Resolution of Line (eV)</th>
</tr>
</thead>
<tbody>
<tr>
<td>122.0</td>
<td></td>
</tr>
<tr>
<td>59.57</td>
<td></td>
</tr>
<tr>
<td>32.191</td>
<td></td>
</tr>
<tr>
<td>22.162</td>
<td></td>
</tr>
<tr>
<td>14.37</td>
<td></td>
</tr>
</tbody>
</table>

**Figure 11**

K X-ray spectrum of mercury.

**Figure 12**

Counts/Channel (Relative)

<table>
<thead>
<tr>
<th>Channel Number</th>
<th>Hg Kα1</th>
<th>Hg Kα2</th>
<th>Hg Kβ1</th>
<th>Hg Kβ2</th>
</tr>
</thead>
<tbody>
<tr>
<td>68.894</td>
<td>82.526</td>
<td>90.258</td>
<td>70.821</td>
<td>68.894</td>
</tr>
</tbody>
</table>
Exercise (c)

Determine the experimental energies of the materials that were fluoresced in Procedure 3 above. Fill in data Table III (an example).

Table III
Comparison of Experimental and Theoretical $K_{\alpha}$ and $K_{\beta}$ Energies

<table>
<thead>
<tr>
<th>Sample</th>
<th>Energy (KeV) (Theory)</th>
<th>Measured Channel</th>
<th>Energy (KeV) (Measured)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Ag $K_{\alpha}$</td>
<td>22.104</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Ag $K_{\beta}$</td>
<td>24.987</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Cd $K_{\alpha}$</td>
<td>23.109</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Cd $K_{\beta}$</td>
<td>26.143</td>
<td></td>
<td></td>
</tr>
<tr>
<td>In $K_{\alpha}$</td>
<td>24.139</td>
<td></td>
<td></td>
</tr>
<tr>
<td>In $K_{\beta}$</td>
<td>27.382</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Sn $K_{\alpha}$</td>
<td>25.193</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Sn $K_{\beta}$</td>
<td>28.601</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

Exercise (d)

For the unknown samples determine the energies of the $K$ X-ray groups; and, hence, determine the elements that are present in the sample. Note there is a listing of $K$ X-ray energies in the back of this manual.

Post-Test

2.1 Define internal conversion coefficients.
Computer Programs

(See the Appendix of this manual.)

GAUSS-6 and LINEAR-6 can be used to accurately establish the calibration curve shown in Figure 6. With the aid of these programs, students have been able to determine the energies of K lines for heavy elements to an accuracy of ±50 eV which is quite good for these detectors.

Additional References

For students who wish to use a germanium detector for senior research-type projects such as pollution analysis, etc., the following additional references will be helpful.


Experiment 3

X-Ray Fluorescence Using a Gas Filled Proportional Counter

Objective

To study the basic properties of gas-filled proportional counters for X-ray measurements; to become familiar with the electronics used with proportional counter X-ray measurements; to construct an energy calibration curve; to X-ray fluoresce known and unknown samples in order to determine the characteristic energies of the K X rays from these samples and thus, to identify the unknown elements; to study escape peaks present in X-ray spectra taken with proportional counters and their causes; to measure the efficiency of a proportional counter as a function of X-ray energy; and to identify unknown radioactive sources from their X-ray spectra.

References


Introduction

X-ray fluorescence experiments can be done with rather inexpensive gas-filled proportional counters. In general, the resolution that one finds with a proportional counter is perhaps a factor of 5 or 10 times worse than would be observed with a high-resolution Si(Li) detector as described in Experiment 1. For example (see Figure 1) the resolution of a typical proportional counter is 15% at an X-ray energy of 10 keV, while the corresponding Si(Li) resolution is about 3%. Nevertheless, proportional counters for this application cost around $300 while the corresponding Si(Li) detector costs at least a factor of ten more.
It will be shown that proportional counters can be used to identify elements, provided the samples are chosen such that the elements present in the sample are far enough apart in atomic number to allow separation with the proportional counter. For example, let us assume that our sample contained arsenic and rubidium. The $K_{\alpha}$ line from arsenic is 10.543 keV, and the corresponding line from rubidium is 8.394. These lines are about 1.8 keV apart and our detector will resolve lines which are 1.5 keV apart in energy; and, hence, we would observe two distinct groups.

At a $K_{\alpha}$ energy of 50 keV the proportional counter would have a resolution of about 3.7 keV. If the sample contained holmium whose $K_{\alpha}$ energy is 47.52 keV we could easily see lutecium ($K_{\alpha} = 54.06$ keV) in the same sample.

In summary, the principles of X-ray fluorescence can easily be taught with a gas filled proportional counter and for some applications standard analysis can be made on, for example, pollution samples.
Equipment

1. Thin Window Proportional Counter for X-Ray Applications
2. Proportional Counter High Voltage Power Supply
3. Pre-amplifier for Proportional Counter Application
4. Low Noise Spectroscopy Amplifier
5. Biased Amplifier (optional)
6. Multichannel Analyzer
7. 10 mCi $^{57}$Co Source for Excitation with Lead Shield
8. Radioactive Sources for Calibration as follows: 1 $\mu$Ci, $^{241}$Am; 10 $\mu$Ci, $^{137}$Cs; 10 $\mu$Ci, $^{57}$Co
9. Samples to Fluoresce (for example: copper, zirconium, palladium, and cadmium. These can all be thin foils.)
10. Oscilloscope (optional)

Procedure

1. Set up the electronics as shown in Figure 2 without the biased amplifier. Be careful not to touch the beryllium window on the proportional counter. These windows are usually 0.0125 cm in thickness and can be broken quite easily. Set the high voltage

X-RAY FLUORESCENCE USING PROPORTIONAL COUNTER

![Diagram of X-ray fluorescence setup]

Electronics for proportional counter X-ray measurements.
power supply to the value recommended by the manufacturer for the tube you are using. Remove the $^{57}$Co source shown in Figure 2 and place the 10 $\mu$Ci $^{137}$Cs source about 1 cm from the face of the detector. Adjust the gain of the main amplifier so that the output pulses for the $^{137}$Ba K$_\alpha$ X rays are about 5 volts in amplitude. Since the Ba K$_\alpha$ X rays have an energy of 32.191 keV, the instrument is now roughly calibrated for 60 keV full scale. This is true since transistorized amplifiers usually put out maximum pulses of around 10 volts.

2. Accumulate a spectrum in the multichannel analyzer for a long enough period of time to get good statistics in the Ba K$_\alpha$ group. Readout the multichannel analyzer and replace the $^{137}$Cs source with the 10 $\mu$Ci $^{57}$Co source. Accumulate for a period of time long enough to get good statistics under the 6.4-keV line. Readout the multichannel analyzer. Repeat this same procedure for $^{241}$Am. Figure 3 shows a typical proportional counter spectrum for $^{57}$Co. Figure 4 shows $^{241}$Am with xenon filled proportional counter.

![Proportional Counter X-ray Spectrum from $^{57}$Co](image)

Figure 3
Proportional counter spectrum for $^{57}$Co.
3. Construct a calibration curve by making a plot of energy vs channel number for the sources used in this experiment. Sources other than the ones mentioned above may be available for this calibration. Table I gives information in regard to convenient X-ray sources and corresponding energies.

Table I
Low Energy X-Ray Sources (\(>1\ \mu\text{Ci}\) or greater)

<table>
<thead>
<tr>
<th>X-Ray Source</th>
<th>Main Energies (MeV)</th>
</tr>
</thead>
<tbody>
<tr>
<td>(^{241}\text{Am})</td>
<td>59.6, 26.36, 20.77, 17.74, 13.95</td>
</tr>
<tr>
<td>(^{137}\text{Cs})</td>
<td>32.2</td>
</tr>
<tr>
<td>(^{113}\text{Sn})</td>
<td>24.2</td>
</tr>
<tr>
<td>(^{40}\text{Sr})</td>
<td>13.5</td>
</tr>
<tr>
<td>(^{57}\text{Co})</td>
<td>14.4, 6.4</td>
</tr>
<tr>
<td>(^{65}\text{Zn})</td>
<td>8.05</td>
</tr>
<tr>
<td>(^{46}\text{Sc})</td>
<td>4.5</td>
</tr>
</tbody>
</table>
Figure 5 shows an energy calibration curve that was made with $^{57}\text{Co}$ and $^{137}\text{Cs}$. Shown on the same spectrum are the conversion X-ray lines from $^{244}\text{Cm}$. $^{244}\text{Cm}$ has L X-ray conversion lines which result from alpha decay to $^{240}\text{Pu}$.

4. Replace the $^{57}\text{Co}$ source to the configuration exactly as shown in Figure 2 and excite the first sample which was supplied to you by the laboratory instructor. Accumulate a spectrum until the K group has good statistics and readout the multichannel analyzer. Figure 6 shows a pulse height spectrum of a thin piece of cadmium that was used for a standard sample with a xenon filled proportional counter. As expected, the most pronounced high energy group is the cadmium K$_\alpha$ group at 23.1 keV. Figure 7 shows a pulse height spectrum of cadmium with a krypton filled proportional counter.

5. From the calibration curve, determine the energies of the pronounced lines in each spectrum accumulated.

6. Obtain three unknown samples from the laboratory instructor. Accumulate spectra for the three samples and fill in K$_\alpha$ measured in Table II for these samples. Figure 8 shows a spectrum of an old silver quarter that was sandwiched between two pieces of cardboard and used as an unknown. Figure 9
Figure 6
Source excited spectrum of cadmium with a xenon filled proportional counter.

Figure 7
Source excited spectrum of cadmium with a krypton filled proportional counter.
Figure 8
$^{57}\text{Co}$ excited spectrum of a silver quarter.

Figure 9
$^{57}\text{Co}$ spectrum of a modern clad quarter.
Table II
Tabulation of Theoretical and Measured Energies (keV)
for Proportional Counter X-Ray Spectra

<table>
<thead>
<tr>
<th>Sample</th>
<th>$K_{\alpha}$ Theory</th>
<th>$K_{\alpha}$ measured</th>
<th>($K_{\alpha}$-Escape) Theory</th>
<th>($K_{\alpha}$-Escape) Measured</th>
</tr>
</thead>
<tbody>
<tr>
<td>Copper</td>
<td>8.041</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Zirconium</td>
<td>15.746</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Palladium</td>
<td>21.123</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Cadmium</td>
<td>23.109</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Silver</td>
<td>22.104</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Molybdenum</td>
<td>17.443</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Unknown A</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Unknown B</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Unknown C</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

shows a spectrum of a modern clad quarter that was given as an unknown in the same type of cardboard configuration.

Fill in the measured energies in Table II for the samples that you have studied. Note that the entry in Table II marked $K_{\alpha}$ theory is the weighted average of $K_{\alpha 1}$ and $K_{\alpha 2}$. The resolution of the gas filled proportional counter does not allow separation of $K_{\alpha 1}$ and $K_{\alpha 2}$ for the elements listed; and, hence, the average value is used. The position of the escape peaks and other features of the spectrum will be discussed later in this experiment.

7. In the figures that are shown in this laboratory report, xenon and krypton filled proportional counters are used. There is a problem inherent with proportional counters associated with their construction. This problem is mainly one of efficiency in regard to generating an output pulse at the full X-ray energy.

In general, pulses will be produced at incident energies of $E_1$ and $E_0 - E_1$, where $E_0$ is the incident X-ray energy and $E_1$ is the escape energy of the $K_{\alpha}$ X ray, associated with the counting gas. In the proportional counter an X ray comes in and makes
a photoelectric interaction with, say for example, a krypton atom in a krypton filled proportional counter. The proportional counter generates an output pulse whose magnitude is composed of several parts. These are:

\[ P = A + B + C \]

where \( P \) = magnitude of the electrical pulse generated from the proportional counter,

\[ A = \text{the energy of the recoil electron produced by the initial photoelectric interaction,} \]
\[ B = \text{the energy of the X ray produced as a result of the vacancy in the K shell of the krypton atom, and} \]
\[ C = \text{the total energies of other X rays and Auger electrons produced in the resulting rearrangement of the krypton atom.} \]

If the X ray B which is the \( K_\alpha \) X ray for krypton (12.630 keV) is captured in the proportional counter, the pulse \( P \) will generally be the full energy pulse; and, hence, proportional to the incident X ray. (In this discussion it is assumed that the initial X-ray energy from the source is high enough to remove the K electron from krypton.) It is possible, however, that the krypton X-ray energy B will escape from the counter. If this happens, the pulse generated will have an energy \( P = A + C \), which is the incident energy of the X ray minus the \( K_\alpha \) X-ray energy for krypton.

Figure 7 is an excellent example of this phenomenon. The lines that we see are the cadmium \( K_\alpha \) line at 23.1 keV and the escape line which corresponds to the cadmium \( K_\alpha \) energy minus the \( K_\alpha \) line from krypton energy, which gives a peak at 10.5 keV. The cadmium \( L_\alpha \) line at 3.2 keV doesn't suffer from the above problem since the L X rays which would produce the problem with L spectra are so low in energy that they are usually captured.

Figure 6 shows the same cadmium \( K_\alpha \) line measured with a xenon filled proportional counter. The escape peak is not present since the initial X-ray energy is not great enough to produce a vacancy in the K shell of the xenon atom. The interactions were in the L shell of xenon; and, hence, no K escape peak. The chromium peak shown in Figure 6 results from the iron and chromium bricks that were used to shield the \( ^{57}\text{Co} \) excitation source. The \( ^{57}\text{Co} \) 14.4 keV gamma line shown in Figures 8 and 9 are from coherent scattering in the sample.
Data Reduction

Exercise (a)

From the calibration curve plotted in Procedure 3, determine the resolution of the proportional counter for the 6.4 and 14.4 keV lines from \(^{57}\text{Co}\) and for the 32.2 keV K\(_\alpha\) line from Ba-137.

Exercise (b)

Identify the escape peaks in the spectra and tabulate (K\(_\alpha\)-Escape) in Table II. Fill in the measured and theoretical values for columns 4 and 5 in Table II.

Exercise (c) (optional)

Obtain an unknown radioactive source from the instructor and determine the energies of its X rays. Are there escape peaks? What is the source?

Exercise (d) (optional)

Obtain standard radioactive sources whose activities are known from the instructor. These might be any of the sources listed in Table I. Place the first source at a distance of 3 cm from the face of the detector and count for a period of time long enough to get 4000 counts under the K\(_\alpha\) group of interest. Readout the spectrum and sum under the K\(_\alpha\) group. Continue for the other sources provided by the instructor. Fill in the entries in data Table III.

### Table III

<table>
<thead>
<tr>
<th>Source</th>
<th>Energy (keV)</th>
<th>Photons/sec Theory</th>
<th>Photons/sec Measured</th>
<th>Efficiency (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>(^{57}\text{Co})</td>
<td>6.4</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>(^{57}\text{Co})</td>
<td>14.3</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>(^{65}\text{Zn})</td>
<td>8.05</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>(^{137}\text{Cs})</td>
<td>32.2</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>(^{85}\text{Sr})</td>
<td>13.5</td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>
The theoretical photons/sec listed in column 3 is the value obtained from the standard activity printed on the source. The measured photons/sec is determined by dividing the sum under the Kα group by the time in seconds. The efficiency is determined by taking the ratio of measured activity to theoretical activity. Usually this value is put in percent by multiplying by 100. Plot a curve of efficiency, ε, versus energy.

Post-Test

3.1 Explain single escape and double escape peaks sometimes found in gamma-ray energy spectra.

3.2 What is an Auger electron?

Computer Programs

The programs GAUSS-6 and LINEAR-6 in the Appendix of this manual can be used to find the centroids of the peaks in the proportional counter spectra. The program LINEAR-6 will also give the slope of the calibration curve which is used in the resolution measurement.

Additional References


Experiment 4

Source Excited X-Ray Fluorescence Applied to Environmental and Geochemical Samples (Si(Li) detector)

Objective

To study the applications of source-excited X-ray fluorescence to environmental samples and geochemical specimens; to study the effectiveness of various excitation sources in stimulating K fluorescence for different ranges of elements; to calibrate a Si(Li) detector using standard sources and with this calibration curve to determine the unknown elemental composition of the environmental and geochemical samples; to investigate the qualitative and quantitative sensitivities of the method.

References


Introduction

It will be assumed that the student is familiar with the basic operations of the Si(Li) detector. X-ray fluorescence with a Si(Li) detector has wide applicability for "trace analysis" in the fields of biology, chemistry, physics, geology industrial problems, and the whole complex field of pollution analysis. Under the best of conditions at this point in time, the technique is sensitive to
approximately one part in a million. The technique, therefore, doesn't even compete in sensitivity with neutron activation analysis which is sensitive for many elements down to the nanogram region. What it does offer, however, is speed. It is possible to determine, as you will see, as many as twenty elements simultaneously from some samples in a time period as short as ten minutes. If you will take a piece of ordinary filter paper and place it over the hose of a vacuum cleaner and let the machine run for two hours in a metropolitan area, you will quickly convince yourself that, for some samples, nanogram sensitivities are not necessary.

The electronics and physical arrangement that will be used for this experiment is shown in Figure 1. The sample can be a solid, liquid, or gas. Gas samples offer a special problem and require a chamber that fits over the top of the detector. At these low energies of excitation (Below 25 keV) the photoelectric process is the most important mechanism by which X-rays are generated in the sample. Essentially, what happens is, a photon from the source comes in and makes a photoelectric interaction with one of the target atoms. During the process a K or L electron is removed. Let's assume it is a K electron since that is what happens most of the time. The K vacancy is quickly filled by an outer electron falling in to fill the void. The most probable thing to happen (see Figure 2) is for an L-III orbital electron to fall into the K vacancy. When this happens, we see the so-called \( K_{\alpha 1} \) X ray. The \( K_{\alpha 1} \) X ray occurs when an L-II orbital falls into the vacancy. Figure 2 shows some of the other possibilities that exist with both K and L initial vacancies. An observer usually sees (for K excitation) the \( K_{\alpha} \) and \( K_{\beta} \) X rays; and the ratio of \( K_{\alpha} \) to \( K_{\beta} \) is usually about seven.

Figure 1
Electronics for X-ray fluorescence with a Si(Li) detector.
In pollution samples, identification is usually made in terms of the $K_a$ line. However, sometimes the $K_a$ line is hidden under another peak and under these conditions, the $K_b$ line can be used. The resolutions of Si(Li) detectors are in general good enough ($\sim 200$ eV at 6 keV) that both the $K_a$ and $K_b$ lines will be seen for most elements that are present in the sample.

At this point the other problem that we must address ourselves to is what excitation source is best suited for the range of elements that are to be studied in a sample. Figure 3 shows a plot on log-log graph paper of the excitation cross sections of several elements as a function of incident photon energy. From this figure, it can rather easily be seen that the best of all possible excitation sources for an element $A_i$ is one whose photon energy is just slightly greater than the absorption edge for the element $A_i$. The absorption edges are the $K_{ab}$ values listed in Appendix V of this manual. (Critical Absorption and Emission Energies of the Elements.) For example, the excitation cross section of aluminum is about $2 \times 10^3$ barns/atom at its absorption edge (1.559 keV); however, it is only $1 \times 10^3$ barns/atom at an incident energy of 7.5 keV. The probability of producing aluminum X rays has therefore fallen off a factor of 200 in going from a source whose photon energy is 1.6 keV to one that has an
incident energy of 7.5 keV. Table I lists a number of standard radioactive sources and the range of elements for each that can be stimulated to K fluorescence.

<table>
<thead>
<tr>
<th>Radioisotope</th>
<th>Source Activity</th>
<th>Half-Life</th>
<th>Radiation Energy</th>
<th>Range of Elements Stimulated to K Fluorescence</th>
</tr>
</thead>
<tbody>
<tr>
<td>$^{55}$Fe</td>
<td>50 mCi</td>
<td>2.6 Y</td>
<td>5.9 keV</td>
<td>Na - V</td>
</tr>
<tr>
<td>$^{109}$Cd</td>
<td>5 mCi</td>
<td>1.29 Y</td>
<td>(88 keV), 22 keV</td>
<td>Ti - Ru</td>
</tr>
<tr>
<td>$^{241}$Am</td>
<td>25 mCi</td>
<td>458 Y</td>
<td>60 keV</td>
<td>Fe - Tm</td>
</tr>
</tbody>
</table>
Equipment

1. Lithium Drifted Silicon X-Ray Detector and Pre-amplifier (resolution <200 eV)
2. Detector Bias Supply
3. Low Noise Spectroscopy Amplifier
4. Multichannel Analyzer (~500 channels)
5. 25 mCi $^{109}$Cd (excitation source)
6. 25 mCi $^{55}$Fe (excitation source)
7. 50 mCi $^{241}$Am (excitation source)
8. Calibration Sources as follows (all should be standard sources ±10% activity):
   a) 10 µCi $^{57}$Co
   b) 1 µCi $^{241}$Am
   c) 10 µCi $^{54}$Mn
   d) 10 µCi $^{51}$Cr
9. Environmental and Geological, etc., Samples to be Studied.

Procedure

1. Set up the electronics as shown in Figure 1. Set the bias voltage to the value recommended by the detector manufacturer. Place the 10 µCi $^{57}$Co source at exactly the location where the samples will be under fluorescing conditions (note the $^{109}$Cd excitation source has been removed for this part of the experiment). Adjust the gain of the system so that the 14.36 keV peak is about mid-scale on your analyzer. Figure 4 shows how this would look on a 400 channel analyzer. The system is now roughly calibrated for 28 keV full scale. Accumulate a timed spectrum for a period of time long enough to obtain ~2000 total counts under the 14.36 keV group. Readout the multichannel and erase.

2. Place the 1 µCi $^{241}$Am source at the same distance as in 1 and accumulate a spectrum for a long enough period of time to get good statistics in all of the pronounced $^{241}$Am peaks. Repeat the same timed measurement for the $^{54}$Mn and $^{51}$Cr sources. From the multichannel readouts, fill in the peak channel information in Table II.

3. Remove the calibration source and place the first environmental sample to be studied in the sample holder. Place the $^{109}$Cd excitation source in its proper position. Fluoresce the sample...
Equipment

1. Lithium Drifted Silicon X-Ray Detector and Pre-amplifier (resolution <200 eV)
2. Detector Bias Supply
3. Low Noise Spectroscopy Amplifier
4. Multichannel Analyzer (~500 channels)
5. 25 mCi $^{109}$Cd (excitation source)
6. 25 mCi $^{55}$Fe (excitation source)
7. 50 mCi $^{241}$Am (excitation source)
8. Calibration Sources as follows (all should be standard sources ±10% activity):
   a) 10 μCi $^{57}$Co
   b) 1 μCi $^{241}$Am
   c) 100 μCi $^{54}$Mn
   d) 10 μCi $^{51}$Cr
9. Environmental and Geological, etc., Samples to be Studied.

Procedure

1. Set up the electronics as shown in Figure 1. Set the bias voltage to the value recommended by the detector manufacturer. Place the 10 μCi $^{57}$Co source at exactly the location where the samples will be under fluorescing conditions (note the $^{109}$Cd excitation source has been removed for this part of the experiment). Adjust the gain of the system so that the 14.36 keV peak is about mid-scale on your analyzer. Figure 4 shows how this would look on a 400 channel analyzer. The system is now roughly calibrated for 28 keV full scale. Accumulate a timed spectrum for a period of time long enough to obtain ~2000 total counts under the 14.36 keV group. Readout the multichannel and erase.

2. Place the 1 μCi $^{241}$Am source at the same distance as in 1 and accumulate a spectrum for a long enough period of time to get good statistics in all of the pronounced $^{241}$Am peaks. Repeat the same timed measurement for the $^{54}$Mn and $^{51}$Cr sources. From the multichannel readouts, fill in the peak channel information in Table II.

3. Remove the calibration source and place the first environmental sample to be studied in the sample holder. Place the $^{109}$Cd excitation source in its proper position. Fluoresce the sample
for a period of time long enough to obtain reasonable statistics in the peaks of interest. Figure 5, for example, shows a dirt sample that was taken near a busy road. Readout the multichannel. Repeat for other environmental or chemical samples. Be sure to run long enough for each sample to get good statistics under the peaks of interest.

![X-ray fluorescence of a dirt sample taken near a busy road.](image)

Figure 5

X-ray fluorescence of a dirt sample taken near a busy road.

4. In order to determine the number of milligrams of a given element in the sample, it is frequently convenient to compare that X-ray line to a standard of a similar matrix which the student prepares in the chemistry laboratory. For example, Figure 6 shows a plot of K X-ray counts per minute per 1% element in the matrix. The sample used could be considered infinitely thick for this application. The matrix used was CaCO₃. Figures 7 and 8 show spectra of a lead standard that was prepared by coprecipitating lead with calcium (CaC₂O₄). The lead concentrations were then determined by atomic absorption.
Figure 6

X-ray CPM/1% concentration of the element in the matrix.

Figure 7

Standardization curve made with Pb Lα.
5. Remove the $^{109}\text{Cd}$ source and place the $^{55}\text{Fe}$ source in the excitation source position. Fluoresce each of the same environmental samples with $^{55}\text{Fe}$. Remember $^{55}\text{Fe}$ is good for elements from sodium up to vanadium. Accumulate each spectrum for a period of time long enough to obtain reasonable statistics in the peaks of interest. For example, Figure 9 shows ordinary cigarette ash that has been fluoresced with $^{55}\text{Fe}$. If a vacuum chamber is available for exciting samples with $^{55}\text{Fe}$ in vacuum, a profound effect can be observed because of the attenuation offered by the air. Also the argon K line from air will show up for samples studied in air. To illustrate this point, Figure 10 shows a silicon sample that was fluoresced for 20 minutes in air. (Note the argon line from air.) Figure 11 shows the same data taken under vacuum conditions.

Data Reduction

Exercise (a)

From the data in Table II, plot an energy versus channel number curve for $^{57}\text{Co}$, $^{54}\text{Mn}$, $^{241}\text{Am}$, and $^{51}\text{Cr}$. Figure 12 shows a similar curve that was taken for K lines in this same energy calibration range.
Figure 9

X-ray fluorescence spectrum of ordinary cigarette ash.

Figure 10

X-ray fluorescence spectrum of silicon in air (\(^{55}\text{Fe}\) source).
Determine the slope of the calibration curve and measure the resolution of the $^{55}$Fe, $^{54}$Mn, and $^{57}$Co lines in Table II.

Exercise (b)

For each line in Table II, sum under the peak of interest and divide by the corresponding time to obtain the measured photons/sec entry in Table II. From the decay schemes for the isotopes used and the absolute disintegration rates tabulated for these standard sources, calculate the theoretical number of photons/sec for each
line in Table II. (Note, in Appendix IV of this manual, there is a tabulation of the latest photon decay information for some of the more commonly used X-ray and gamma calibration sources.) The absolute efficiency for the detector is defined as the measured photons/sec divided by the actual number of photons/sec that the source emits. (Be sure to make any necessary half-life corrections required on calibration sources.) This quantity is called [e]. Calculate [e] for the values in Table II. Plot a curve of efficiency versus energy for the detector. Figure 13 shows a similar curve for a silicon detector and various window thicknesses. Since you are not correcting your data for geometry and the one over R² intensity fall off, your maximum efficiency will probably be about 10%. You can convert your data to a curve similar to Figure 13 if you make the necessary corrections.

![Figure 13](image)

Efficiency plot for a Si(Li) detector.

Exercise (c)

From the calibration curve, identify the major peaks in your environmental or chemical samples for the ¹³¹I excitation. If the samples are thin and uniform, you may also wish to determine the amount of each element that is present in your sample. Figure 14 shows a spectrum of a dried water sample that was taken at the exit water channel of a sewer water treatment plant in Nashville. Since the sample was quite thin, quantitative information could
also have been extracted from the multichannel readout. Figure 15 shows an air pollution sample that was obtained from the Air Quality Control Board in Nashville. Again since it was a thin sample, the techniques discussed in Procedure 4 are applicable for extracting absolute information in regard to the peaks.

Exercise (d)

Repeat Exercise (c) for all of the data taken with your samples and the $^{55}$Fe source. Figure 16 shows a thin sample of powdered coal. Since the sulfur component in coal seems to give the largest pollution problem at coal-fire energy generation plants, this technique offers a good method of monitoring sulfur content in coal. Figure 17 shows some fly ash that was collected from the Bull Run coal-fire generating plant in Oak Ridge, Tennessee. Note the silicon, potassium, calcium, and titanium is present in the sample.
Figure 15
X-ray fluorescence spectrum of an air pollution filter.

Figure 16
X-ray fluorescence spectrum of soft coal.
Figure 17

X-ray fluorescence spectrum of fly ash from a coal-fire electric generating plant.

as in the original coal sample. The sulfur is not, since it comes out of the stack as SO and SO2.

Exercise (e)

Use the technique discussed in Procedure 6 to extract absolute numbers in milligrams for the elements present in your samples. Figures 18, 19, and 20 show other spectra that are of general interest in pollution analysis.

Post-Test

4.1 A 5 μCi 141Am source is placed 10 cm from a Si(Li) detector with an active surface area of 3 mm2. Using Figure 13 and Appendix IV, determine the number of counts an observer would expect to find beneath the Lα, Lβ, Lγ X-rays peaks and the 26.4 keV and 59.54 keV gamma-ray peaks after a 10 minute counting period. Neglect edge effects and attenuation in air and Be windows.
Air Pollution Sample
$^{109}$Cd XRF (20 min)

Figure 18
X-ray fluorescence spectrum of air pollution filter ($^{109}$Cd excitation source).

Final Water Sample
$^{55}$Fe XRF (20 min)

Figure 19
X-ray fluorescence spectrum of exit water from treatment plant ($^{55}$Fe source).
The Mn Kα & Kβ are from the 55Fe Source

![Graph showing various peaks including Mn Kα, Mn Kβ, Ca Kα, Ca Kβ, Ti Kα, Ar Kα, and a note about channel number and counts per channel.]

Figure 20

X-ray fluorescence of shavings from an automobile tire (55Fe excitation source).

Computer Programs

GAUSS-6 can be used to find the centroids of all calibration and environmental spectra. LINEAR-6 can be used to find the slope of the calibration curve and for identifying all lines in the environmental spectra.

Additional References


Experiment 5

Heavy Element Analysis of Pollution Samples by X-Ray Fluorescence with a Ge(Li) Detector

Objective

To study the application of source excited X-ray fluorescence with a Ge(Li) detector; to establish an efficiency curve for this process; to investigate heavy element $Z > 32$ concentration in pollution and chemical samples.

References


Introduction

In this experiment it will be assumed that the student is already familiar with Ge(Li) detectors and the techniques of basic X-ray fluorescence experiments. He may also wish to read the introduction of Experiment 4 which is a rather detailed discussion of some of the parameters associated with X-ray fluorescence. Most of the information in Experiment 4 is applicable in this experiment.

The main differences between the Si(Li) and Ge(Li) detectors for X-ray fluorescence are the efficiency and resolution. In general, Si(Li) devices have resolutions of 200 eV at 6 keV while Ge(Li) would show perhaps 350 eV at this energy. However, the Ge(Li) devices are much more efficient for photons in the energy range from 30 to 100 keV than the Si(Li). The reason for this improved efficiency comes from the fact that photons at these energies usually interact by the photoelectric process. The cross section for the photoelectric process varies like $Z^5$, where $Z$ is the atomic number. The $Z$ for silicon is 14 and for germanium it is 32. The ratio of these two numbers raised to the fifth power is 62.22 which is roughly the ratio of the cross sections.
The arguments made in Experiment 4 about the cross section for excitation being greatest just above the absorption edge are equally valid for samples that will be studied in this experiment. The only difference is that in general with Ge(Li) systems, one is interested in higher Z elements in the sample; and, hence, the exciting source will have a higher energy than the ones used for Si(Li) spectroscopy. In this experiment, we are going to use $^{241}$Am and $^{57}$Co as excitation sources. For $^{241}$Am it will be the 59.6 keV gamma that does most of the excitations in our samples. In $^{57}$Co the 122 keV line is the best candidate for excitation.

**Equipment**

1. Lithium Drifted Germanium X-Ray Detector with a Resolution $\approx 350$ eV
2. Detector Bias Supply
3. Low Noise Spectroscopy Amplifier
4. Multichannel Analyzer ($\approx 500$ channels)
5. Excitation Sources as follows (license required):
   a) 25 mCi $^{241}$Am
   b) 25 mCi $^{57}$Co
6. Calibration Sources as follows:
   a) 10 $\mu$Ci $^{57}$Co standard activity $\pm 10\%$
   b) 1 $\mu$Ci $^{241}$Am standard activity $\pm 10\%$
   c) 5 $\mu$Ci $^{137}$Cs standard activity $\pm 10\%$
   d) 2 $\mu$Ci $^{109}$Cd standard activity $\pm 10\%$
7. Pollution or Chemical Samples to be Studied

**Procedure**

1. Set up the electronics as shown in Figure 1. Do not put in the $^{57}$Co excitation source. The system first has to be calibrated.
2. Turn on the bias voltage to the detector and adjust it to the recommended value. Place the 1 $\mu$Ci standard source at the position where the sample will be when fluorescing pollution samples. Adjust the gain of the system until the 59.6 keV line from $^{241}$Am falls about mid-scale on the analyzer.
3. Collect a timed spectra for a period of time which is sufficient to give reasonable statistics in the pronounced lines of $^{241}$Am shown in Tab. Readout the multichannel.
Figure 1
Electronics for X-ray fluorescence with a Ge(Li) detector.

Table I
Absolute Calibration for a Ge(Li) Detector

<table>
<thead>
<tr>
<th>Nuclide</th>
<th>Photon Energy (keV)</th>
<th>Peak Channel</th>
<th>Photons/sec Measured</th>
<th>Photons/sec Calculated</th>
<th>Efficiency</th>
</tr>
</thead>
<tbody>
<tr>
<td>$^{57}$Co</td>
<td>6.40</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>$^{57}$Co</td>
<td>14.36</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>$^{57}$Co</td>
<td>122.0</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>$^{57}$Co</td>
<td>136.3</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>$^{137}$Cs</td>
<td>32.191</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>$^{109}$Cd</td>
<td>87.70</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>$^{113}$Cd</td>
<td>24.942</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>$^{241}$Am</td>
<td>26.36</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>$^{241}$Am</td>
<td>59.57</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>
4. Collect a timed spectrum for the $^{57}\text{Co}$ standard source. Figure 2 shows a typical spectrum for $^{57}\text{Co}$. Readout the multichannel. Repeat for the standard $^{137}\text{Cs}$ and $^{109}\text{Cd}$ sources. From the readouts determine the peak channels for the entries in Table I.

![Image of a spectrum showing peak channels for $^{57}\text{Co}$]

**Figure 2**
Spectrum of $^{57}\text{Co}$ with a Ge(Li) detector.

5. Remove the calibration source and place the $^{57}\text{Co}$ excitation source in its proper position. Place the first pollution sample to be studied in the proper location. Fluoresce this sample for a period of time long enough to get reasonable statistics in the peaks of interest. Readout the multichannel and repeat this same measurement for the rest of your samples. Figure 3 shows a typical spectrum that was measured in an exercise similar to this.

6. Replace the $^{57}\text{Co}$ excitation source with the $^{241}\text{Am}$ excitation source. Repeat the measurements made in 5 for all of the samples. Figure 4 shows a typical spectrum of a sample that was fluoresced with photons from $^{137}\text{Cd}$.

7. In trying to get absolute numbers for the peaks of interest, it is most convenient to use thin samples. If the samples are too thick, then attenuation problems must be considered. If, however, the samples are thin, then the number of micrograms/cm$^2$ of the element of interest can be determined simply by comparing
Figure 3
X-ray fluorescence spectrum of a water sample.

Air Pollution Sample

Figure 4
X-ray fluorescence spectrum of an air pollution sample.
that element to a standard which you make up in the laboratory. For example, let us assume that we are interested in the lead peaks (K lines) that appear in an air pollution spectrum. Most air pollution spectra collected on filter paper with a vacuum system can be considered thin for the K lines of lead. Then, all we have to do is to dry a known concentration of a lead solution onto a piece of clean filter paper. For example, workers in the state of California dried a mother solution of lead acetate onto these above mentioned filters. Later analysis indicated that their various samples were accurate to ±5%. Table II shows the net count rate that was observed as a function of lead concentration in μg/cm².

### Table II

**Calibration Results from Lead Acetate Standards**

<table>
<thead>
<tr>
<th>Concentration (μg/cm²)</th>
<th>Net Count Rate (counts/min)</th>
</tr>
</thead>
<tbody>
<tr>
<td>100.0</td>
<td>12,403</td>
</tr>
<tr>
<td>50.0</td>
<td>6,328</td>
</tr>
<tr>
<td>10.0</td>
<td>1,357</td>
</tr>
<tr>
<td>5.0</td>
<td>769</td>
</tr>
<tr>
<td>2.0</td>
<td>323</td>
</tr>
<tr>
<td>1.0</td>
<td>167</td>
</tr>
<tr>
<td>0.5</td>
<td>78</td>
</tr>
</tbody>
</table>

Following the general guidelines of the above discussion, prepare standards of known concentration solutions for drying onto filter paper. Prepare enough standards to bracket the concentration level of the groups of interest in one of your spectra. Construct a data table similar to Table II for your samples.

**Data Reduction**

**Exercise (a)**

Plot a calibration curve for the peak channel data that was collected in Table I. Figure 5 shows a typical curve that was taken with a Ge(Li) detector.
From the calibration curve, determine the slope of the line and calculate the resolution of the following lines: 122 keV $^{57}$Co, 87.7 keV $^{109}$Cd, and 59.57 $^{241}$Am.

Exercise (b)

From the timed spectra collected in Procedures 3 and 4, determine the sums under the peaks listed in Table I. Divide these sums by the proper times and thus determine the measured photons/sec for each of these standard sources. Enter these values in their proper places in Table I.

Exercise (c)

Calculate the number of photons/sec that are emitted from each of the standard sources in Table I. Sometimes this information is printed on the standard. If not, it can be determined from the activity of the source and known information in regard to the decay scheme for that isotope. In the appendix of this manual is a tabulation of certain disintegration data for many of the standard calibration sources for X-ray and gamma-ray spectroscopy. Fill in the calculated number of photons/sec for each of the entries in Table I.
Exercise (d)

The efficiency of the detector (ε) is the ratio of the measured photons/sec to the calculated photons/sec. Determine this number for your data and record the proper value for each of the entries in Table 1. Plot a curve of ε versus the photon energy in keV.

Exercise (e)

For each of your pollution samples, determine what elements are present in the sample from your calibration curve. Do this for both $^{57}$Co and $^{241}$Am excitation.

Exercise (f)

With your laboratory instructor's assistance, standardize some of the peaks in accordance with the method outlined in Procedure 7.

Computer Programs

The centroids of all of the peaks measured in this experiment can be found with the program GAUSS-6. LINEAR-6 can be used to identify what lines are present in each pollution spectra.

Additional References

1. J. L. Cate, Jr., Determination of Lead in Air Sample Filters by X-Ray Fluorescence, Lawrence Radiation Laboratory Chemistry Report No. TID-4500, UC-4, UCRL-51038, Livermore, California 94550.


Experiment 6

Tube Excited X-Ray Fluorescence

Objective

To study the parameters associated with X-ray fluorescence measurements where the exciting photons are generated with an X-ray tube; to determine an energy calibration curve with X rays of known energy produced from a variety of elements; to use the calibration curve to determine what unknown elements are present in the pollution, environmental or biological samples; to stand-

ardize measurements with known concentrations of the groups of interest.

References


Introduction

In a few of the earlier experiments, we outlined the use of source excited X-ray fluorescence for problems such as pollution analysis and the geochemical analysis of rock samples. Under the very best experimental conditions, source excited X-ray measurements are good to one part in a million. In other words, with this technique and under the best conditions, we can study microgram concentration in gram samples. However, it was also pointed out that for many applications this kind of sensitivity is adequate. In general, tube excited X-ray fluorescence is about a factor of ten better.

The systems that have been manufactured specifically for this application are in general designed to maximize this sensitivity.

In this discussion there is not time to go into the various systems and their operational parameters. This information can be obtained by reading References 1-4. In particular, Reference 1 is...
a report that carefully outlines the operational parameters of a low powered X-ray tube for this application. In fact, many of the illustrations in this experiment were taken from Reference 1. The companies that manufacture tube-excited systems can furnish additional engineering parameters for their various units.

It is possible under some conditions to convert old X-ray diffraction units into fluorescent spectrometers. In theory, all one has to do is extract the X-ray beam and let it impinge on the sample to be studied. In practice, one has to worry about scattering problems, tube to sample distances, efficiency, etc. It is, however, possible to convert these units to fluorescing devices. Several places have made these conversions with satisfying results.

The additional sensitivity of the tube excited system can be justified quite easily, if one looks at the number of exciting photons per unit time for a tube and a source; for example,

1) 100 mCi source gives $3.7 \times 10^9$ photons/sec

2) 100 $\mu$amp current tube gives $1 \times 10^{12}$ photons/sec.

The additional advantage of the tube comes from the fact that, in theory at least, one can set the accelerating voltage for the tube or choose the X-ray tube target to maximize the sensitivity of the system. In Experiment 4 the arguments were presented in regard to the yield, absorption edge, and incident photon energy. The student should go back and read the experiment.

For an unfiltered X-ray tube the Bremsstrahlen and the characteristic X rays from the target are both effective in fluorescing the target. Figure 1 shows what the spectrum looks like for a molybdenum X-ray tube and an accelerating voltage of 50 keV. In many cases filtering is desirable. For example, if one was studying rubidium ($K_{ab} = 15.20$ keV) the tube used in Figure 2 would be ideal from almost every aspect.

![Figure 1](attachment:figure1.png)

Typical unfiltered spectrum from a molybdenum target (X-ray tube).
Figure 2 shows a molybdenum spectrum that has been collected from a transmission anode tube. In this figure, the filtering is done by the anode material.

This introduction has been more of an overview. We have listed enough references so that even if a university doesn't have a tube excited X-ray fluorescence system, a student would be able to read the references and turn in a nice senior project as to the details of this strong analytic technique.

Equipment

1. Complete Tube Excited X-Ray Fluorescence Analyzer and Readout (see instructor for recommended operating parameters)

2. Standard Materials (reagent grade) to be Used for Calibration (thick samples)

3. Pollution, Environmental, or Biological Samples to be Studied

Procedure

1. Place a piece of manganese metal in the excitation position. Fluoresce it for a period of time long enough to get reasonable statistics under the $K_\alpha$ and $K_\beta$ peaks (see Figure 3).

2. Repeat the measurements made in 1 for titanium, copper, arsenic, and yttrium. Fill in the channel locations in Table I. Record the time for each run. Make a calibration plot of Energy (keV) versus channel number (see Figure 4).
Table I
Energy Calibration Data for a Tube Excited System Si(Li)

<table>
<thead>
<tr>
<th>Sample</th>
<th>Photon Energy (keV)</th>
<th>Peak Channel</th>
<th>Resolution (eV)</th>
<th>Counting Rate counts/min</th>
</tr>
</thead>
<tbody>
<tr>
<td>Mn Kα1</td>
<td>5.898</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Mn Kβ1</td>
<td>6.409</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Ti Kα1</td>
<td>4.510</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Ti Kβ1</td>
<td>4.931</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Cu Kα1</td>
<td>8.047</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Cu Kβ1</td>
<td>8.904</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>As Kα1</td>
<td>10.543</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>As Kβ1</td>
<td>11.725</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Y Kα1</td>
<td>14.957</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Y Kβ1</td>
<td>16.736</td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>
3. Pollution samples or other samples can now be studied. Run each sample for a long enough time to get reasonable statistics in the peaks of interest. Figure 5 shows a typical air pollution sample.

4. In order to obtain quantitative information about a particular group, the procedures outlined in Experiments 4 and 5 may be used at this point.
Data Reduction

Exercise (a)

From the peak channel information in Table I, construct an energy calibration curve. Determine the slope of the calibration curve. From the slope of the calibration curve and the FWHM for each peak listed in Table I, determine the resolution of each of these lines. Fill in this entry in Table I.

Exercise (b)

From Table I plot a curve of resolution versus energy for your system. For each peak listed in the table, sum under the group of interest and divide by the corresponding time and thus determine the counting rate for a given sample under the conditions used. Fill in this entry in Table I.

Exercise (c)

For the pollution, environmental, or biological samples, determine what elements are present in the samples. Figure 6 shows a spectrum that was obtained from a camellia leaf with a molybdenum tube. Figure 7 shows a freeze dried blood sample. Figure 8 is a spectrum of a piece of dried swordfish. The mercury lines in the spectrum correspond to concentrations of the order of 3 parts per million.

Exercise (d)

In order to get quantitative information, the samples should be thin as in Experiments 4 and 5. Make up standards with known concentrations and compare the groups of interest with standards of these materials. Table II which was taken from Reference 2 shows a good agreement between tube excited X-ray fluorescence and NBS data for a steel sample.

<table>
<thead>
<tr>
<th>Element</th>
<th>X-Ray Fluorescence %</th>
<th>NBS</th>
</tr>
</thead>
<tbody>
<tr>
<td>Ti</td>
<td>0.37</td>
<td>0.36</td>
</tr>
<tr>
<td>Cr</td>
<td>19.50</td>
<td>18.69</td>
</tr>
<tr>
<td>Mn</td>
<td>1.43</td>
<td>1.28</td>
</tr>
<tr>
<td>Ni</td>
<td>10.92</td>
<td>10.58</td>
</tr>
</tbody>
</table>
Table III (also taken from Reference 2) shows a similar comparison that was made on a plant spectrum for X-ray fluorescence and neutron activation analysis.

Post-Test

6.1 Discuss how X rays are produced in a tube-excited system or an X-ray machine.

Computer Programs

GAUSS-6 can be used to find the centroids of the tube excited fluorescent lines. LINEAR-6 can be used to do a least squares fit to the standard samples that were used for the calibration curve.
Figure 7
Tube excited spectrum of freeze dried blood.

Figure 8
Tube excited spectrum of dried swordfish sample.
### Table III

Analysis of Plant Spectrum

<table>
<thead>
<tr>
<th>Element</th>
<th>X-Ray Fluorescence</th>
<th>Neutron Activation</th>
</tr>
</thead>
<tbody>
<tr>
<td>Ti</td>
<td>121 ppm ± 5</td>
<td>&lt; 0.01%</td>
</tr>
<tr>
<td>Cr</td>
<td>26 ppm ± 1</td>
<td>23.8 ppm ± 0.9</td>
</tr>
<tr>
<td>Mn</td>
<td>60 ppm ± 2</td>
<td>49.3 ppm ± 1.4</td>
</tr>
<tr>
<td>Fe</td>
<td>0.186% ± 0.002</td>
<td>0.201% ± 0.006</td>
</tr>
<tr>
<td>Ni</td>
<td>8 ppm ± 1</td>
<td>13.8 ppm ± 3.0</td>
</tr>
<tr>
<td>Cu</td>
<td>21 ppm ± 1</td>
<td>-. -</td>
</tr>
<tr>
<td>Zn</td>
<td>80 ppm ± 1</td>
<td>84 ppm ± 8</td>
</tr>
<tr>
<td>Br</td>
<td>48 ppm ± 1</td>
<td>42 ppm ± 1</td>
</tr>
<tr>
<td>Rb</td>
<td>7 ppm ± 1</td>
<td>7.0 ppm ± 1.4</td>
</tr>
<tr>
<td>Sr</td>
<td>97 ppm ± 2</td>
<td>236 ppm ± 66</td>
</tr>
<tr>
<td>Pb</td>
<td>206 ppm ± 3</td>
<td>-. -</td>
</tr>
</tbody>
</table>

**Additional References**


ANSWERS TO QUESTIONS AND PROBLEMS

Pre-Test Answers

1.1 \( c = \lambda f \)

\[
f = \frac{c}{\lambda} = \frac{3 \times 10^{10} \text{ cm/sec}}{10^{-8} \text{ cm}} = 3 \times 10^{18} \text{ oscillations/sec}
\]

1.2 \( E = hf \)

Where \( h \) is Planck's constant and is equal to \( 6.625 \times 10^{-27} \text{ erg-sec} \)

\[ E = (6.625 \times 10^{-27} \text{ erg-sec})(3 \times 10^{18} \text{ oscillations/sec}) \]

\[ = 19.8 \times 10^{-9} \text{ erg} \]

Converting to units of keV

\[ E = (19.8 \times 10^{-9} \text{ erg}) \left( \frac{1}{1.6 \times 10^{-12} \text{ erg}} \right) \]

\[ = 12.4 \times 10^{3} \text{ eV} \]

\[ = 12.4 \text{ keV} \]

2.1 An electron volt is the kinetic energy acquired by an electron which is subjected to a potential difference of 1.0 volt.

2.2 \( \text{keV} = 10^{3} \text{ eV} \)

\( \text{MeV} = 10^{6} \text{ eV} \)

\( \text{GeV} = 10^{9} \text{ eV} \)

3.1 Postulates of Bohr theory of the atom

a) An electron bound to an atom moves in a circular orbit about the nucleus under the influence of the coulomb.
attraction between the electron and the nucleus and obeying the laws of classical mechanics.

b) The allowed electronic orbits are those for which the orbital angular momentum of the electron, \( L \), is an integral multiple of Planck's constant divided by \( 2\pi \).

c) Electromagnetic radiation is emitted if an electron, initially moving in an orbit of total energy, \( E_i \), suddenly changes its motion so that it moves in an orbit of total energy, \( E_f \). The frequency of the emitted radiation is \( f = (E_i - E_f)/h \).

\[ 3.2 \quad E_i - E_f = hf \]
\[ = (6.625 \times 10^{-27}, \text{erg-sec})(8.45 \times 10^{18} \text{ oscillations/sec}) \]
\[ = 56 \times 10^{-9} \text{ erg} \]
\[ = 35 \text{ keV} \]

4.1 The force is given by the familiar coulomb's law for point charges,

\[ F = \frac{1}{4\pi \varepsilon_0} \frac{q_1 q_2}{r^2} \]

where \( q_1 \) and \( q_2 \) are the electronic charges, \( r \) is the distance separating the two electrons and \( \varepsilon_0 \) is the permittivity of free space and has the value \( \varepsilon_0 = 8.854 \times 10^{-12} \text{ coul}^2/\text{n-m}^2 \).

\[ F = \frac{1}{4\pi (8.854 \times 10^{-12} \text{ coul}^2/\text{n-m}^2)} \frac{(1.6 \times 10^{-19} \text{ coul})^2}{(0.05 \text{ m})^2} \]
\[ = 9.2 \times 10^{-26} \text{ n} \]

4.2 The nuclear coulomb barrier height is defined as the point where the coulomb barrier reaches a maximum value. This occurs at the nuclear surface of the atom of zinc. The maximum height of the barrier is given by

\[ B = \frac{(Ze)}{R} \]
where $Z_e$ is the nuclear charge of the zinc atom, $Z_e$ is the incident alpha particle charge, and $R$ is the nuclear radius of zinc.

$$R = 1.2 \times A^{1/3} \text{ fermis}$$

$$= 1.2 \times (64)^{1/3}$$

$$= 4.8 \text{ fermis}$$

Multiplying both the numerator and denominator by Planck's constant divided by $2\pi$ and the speed of light, one obtains:

$$B = \frac{Z_e^2 \varepsilon^2}{R} \frac{\hbar c}{\hbar c}$$

$$= \frac{Z_e^2 \varepsilon^2}{R} \frac{\hbar c}{\hbar c}$$

substituting

$$\frac{\varepsilon^2}{\hbar c} = \text{the fine structure constant}, \alpha = \frac{1}{137}$$

and

$$\frac{\hbar c}{\hbar c} = 197.32 \text{ MeV} - \text{fermis}$$

$$B = \frac{(30)(2)}{(4.8 \text{ fermis})} \frac{1}{137} (197.32 \text{ MeV} - \text{fermis})$$

$$= 18 \text{ MeV}$$

5.1 In a multi-electron atom there can never be more than one electron in the same quantum state.

6.1 $E_{\text{max}} = hf - W$

$$= (6.625 \times 10^{-27} \text{ erg-sec})(10^{16} \text{ cycles/sec}) - 4 \text{ eV}$$

$$= (6.625 \times 10^{-11} \text{ erg})(\text{lev}/1.6 \times 10^{-12} \text{ erg}) - 4 \text{ eV}$$

$$= 41.4 \text{ eV} - 4 \text{ eV}$$

$$= 37.4 \text{ eV}$$
6.2 If the incident photon is completely absorbed by the $e^-$, the conservation of total relativistic energy gives:

$$E_Y + E_{e^-} = E_{e^+}$$

where $E_Y$ can be rewritten

$$E_Y = hf = \frac{hc}{\lambda} = p_Y c$$

and

$$E_{e^-} = \sqrt{p^2c^2 + m^2c^4}$$

$$p_Y c + \sqrt{p^2c^2 + m^2c^4} = \sqrt{p^2c^2 + m^2c^4}$$

$p_i = 0$, since initially the momentum of the electron is equal to zero.

Therefore, we have

$$p_Y c + m_0 c^2 = \sqrt{p_f^2c^2 + m_0^2c^4}$$

By the conservation of momentum,

$$p_Y = p_f$$

Substituting in the above equation for $p_Y$, we obtain

$$p_f c + m_0 c^2 = \sqrt{p_f^2c^2 + m_0^2c^4}$$

Hence, both total relativistic energy and momentum cannot be conserved.

7.1 In electron capture, the nucleus captures a negatively charged atomic electron. The atomic number $Z$ decreases by one and the neutron number $N$ increases by one. The energy available for electron capture by the nucleus is

$$E = \left[\frac{A}{Z^M} - \frac{A}{Z-1}^M\right] c^2$$

In internal conversion, the excitation energy of the nucleus is transferred directly to the electron through the coulomb
interaction between the two. The nucleus de-excites, and the electron is ejected.

7.2 \[ E = M(50\text{V})c^2 - M(50\text{Ti})c^2 - 1.55 \text{ MeV} \]
\[ = (49.9472 \text{ amu})c^2 - (49.9448 \text{ amu})c^2 - 1.55 \text{ MeV} \]
\[ 1 \text{ amu} = 931.162 \text{ MeV} \]
\[ E = 46,508.93 \text{ MeV} - 46,506.70 \text{ MeV} - 1.55 \text{ MeV} \]
\[ = 0.685 \text{ MeV} \]

Post-Test Answers

1.1 The energy of the characteristic X-rays depends on the binding energies of the electrons in the inner shells. With increasing atomic number, Z, these binding energies increase uniformly, due to the increased nuclear charge and are not affected by the periodic changes in the number of electrons in the outer shells.

1.2 A 1.0 curie source has an activity of \(3.7 \times 10^{10}\) disintegrations per second.

\[ 0.1 \text{mCi} = 1 \times 10^{-4} \text{ Ci} \]

Hence, a 0.1 mCi source decays at a rate of \(3.7 \times 10^6\) disintegrations per second.

1.3 To shield the lithium drifted silicon detector from the ring source of radiation.

1.4 Number of counts \(\approx 3000 \text{ counts/channel} \times 6 \text{ channels} \approx 18,000 \text{ counts}\)

1.5 a) \[ \frac{dE}{d\text{c}} = \frac{11,729 \text{ eV} - 4931 \text{ eV}}{214.63 \text{ ch} - 83.17 \text{ ch}} \]
\[ = 51.71 \text{ eV/channel} \]

b) \[ \frac{dE}{d\text{c}} = \frac{14,933 \text{ eV} - 8041 \text{ eV}}{277.41 \text{ ch} - 143.29 \text{ ch}} \]
\[ = 51.40 \text{ eV/channel} \]
1.6  a) \( I_0 \times \frac{1}{(2)^5} = \frac{I_0}{32} \)

b) 5

1.7 Yes.

2.1 An internal conversion coefficient is the ratio of the probability for emitting an electron from a given shell to the probability for emitting a gamma ray.

3.1 Escape peaks may be produced in a gamma-ray energy spectrum if the energy of the incident gamma ray, \( E_\gamma \), is greater than the rest mass energy of two electrons. If the incident gamma ray interacts by means of the pair production process, then, 1.022 MeV of energy is required to produce the electron-positron pair and the remaining photon energy is given to the charged particles as kinetic energy. This kinetic energy is deposited in the detector as the two charged particles lose energy through coulomb interactions and come to rest. The positron and an electron annihilate each other producing two 511 keV photons. If both 511 keV photons escape the detector then a double escape peak of energy, \( E_d \), is produced.

\[
E_d = E_\gamma - 1.022 \text{ MeV}
\]

If one 511 keV photon is detected the energy of the escape peak, \( E_s \), is

\[
E_s = E_\gamma - 0.511 \text{ MeV}
\]

If both 511 keV photons are detected then a full energy peak is observed.

\[
E = E_\gamma
\]

3.2 An Auger electron is an electron ejected from an atom in a radiationless transition. For example, an atom in which a K shell electron has been ejected has a large probability of emitting a \( K \alpha \) X ray as an L shell electron falls into the K shell. If, however, the atom de-excites through the emission of an L shell electron, the radiationless transition is called an Auger transition and the L shell electron ejected is an Auger electron.
4.1 The number of counts an observer would expect to find in the peaks is given by the expression:

\[
N = N_D \cdot T \cdot N_P \cdot \varepsilon \cdot \frac{d\Omega}{4\pi}
\]

where
\(N_D\) = number of disintegrations per second of the 5 μCi 241Am source
\(T\) = total time in seconds
\(N_P\) = number of photons emitted per disintegration (Appendix IV—Table I)
\(\varepsilon\) = efficiency of the Si(Li) detector (Figure 13)
\(d\Omega = \frac{dA}{r^2}\) = solid angle of detector.

\[
N_{\alpha} = (5 \times 10^{-6} \times 3.7 \times 10^{10} \text{ disint/sec})(600 \text{ sec})
\times (0.135 \text{ photons/disint})(1.0)\left(\frac{3 \times 10^{-4} \text{ steridians}}{4\pi}\right)
\]

\(= 358 \text{ photons}\)
\(N_{\beta} = 535 \text{ photons}\)
\(N_{\gamma} = 125 \text{ photons}\)
\(N_{26.4} = 44 \text{ photons}\)
\(N_{59.5} = 76 \text{ photons}\)

6.1 Present-day X-ray tubes produce X rays through the collision of electrons with a heavy material such as tungsten. The electrons are produced by a heated filament and are accelerated to high velocities by means of an electric field before striking the anode material. The X-ray tube is operated at high vacuum in order that the largest number of electrons will impinge upon the anode material. X rays emitted from the anode material are defined into a beam by lead collimators.
INTRODUCTION

Solid-state detectors have been in use for the detection of ionizing radiation for the past two decades. The first use of a p-n junction device as a solid-state detector was by K. G. McKay for alpha particle detection. Since then, their use has revolutionized charged particle detection.

Semiconductor charged particle detectors may be used over a broad range of energies and projectiles from 20 keV electrons to 200 MeV heavy ions. These surface-barrier detectors may be prepared with energy resolution of better than 0.5% which is surpassed only by magnetic spectrometers. The timing characteristics of these detectors (<1 nsec pulse rise time) are good enough to perform fast coincidence experiments and in addition their efficiency is essentially 100% for their active volume.

The module consists of three experiments which may be performed by the student. In the first experiment the student will be introduced to some of the procedures and detectors commonly employed in alpha particle studies. In Experiment 2 the student will be familiarized with the use of solid-state charged particle detectors for conversion electron spectroscopy. In Experiment 3 a thin transmission surface barrier detector is employed to demonstrate "state of the art" nuclear timing techniques.

This introduction is followed by the objectives for the module, a list of prerequisites which the student should have before attempting this module, and a theoretical section which discusses some of the basic concepts of radioactive decay and semiconductor detectors.

OBJECTIVES

To familiarize the student with the use of solid-state charged particle detectors for alpha particle studies and conversion electron spectroscopy.
PREREQUISITES WITH PRE-TEST QUESTIONS AND PROBLEMS

1. Coulomb Force:
   1.1 Determine the magnitude and direction of the coulomb force between two electrons 5 cm apart.
   1.2 What is the nuclear coulomb barrier height in MeV for a 5 MeV proton incident upon an atom of copper (A = 65).

2. Electron Volt (eV), keV, MeV:
   2.1 How is an electron volt defined?
   2.2 How is the eV related to the multiples keV, MeV, GeV?

THEORY

Natural Radioactivity

Naturally occurring radioactive isotopes emit alpha, beta, or gamma radiation. If an alpha particle is emitted by the nucleus of an atom, the atom is transformed into a new atom which has its atomic mass, A, decreased by 4 units and its atomic number, Z, decreased by 2 units. If a beta particle is emitted from a nucleus of atomic number, Z, the atomic number of the new atom formed is Z + 1 and the atomic mass remains the same. Two genetically related nuclei are called isobars. Gamma radiation is sometimes associated with either alpha or beta decay of a radioactive nucleus.

Almost all the radioactive nuclei occur within the range of atomic numbers Z = 81 to Z = 92. These elements have been grouped into three genetically related series: the uranium-radium series, the thorium series and the actinium series. A very long-lived isotope begins each series while one of the stable lead or bismuth isotopes ends each.

Rate of Radioactive Decay

The decay rate of a particular radioactive material is a constant that is independent of physical and chemical conditions. The average number of atoms, dN, that will decay in a small time interval, dt, is proportional to the number of atoms, N, present at the time, t.

\[-dN = \lambda N dt\]
The constant of proportionality, $\lambda$, is the decay constant for that particular radioactive isotope. Integrating, one obtains

$$N = N_0 e^{-\lambda t},$$

where $N_0$ is the number of atoms present at time $t = 0$. Hence, the number of atoms of a given radioactive substance decreases exponentially with time assuming no new atoms are introduced.

At the end of a certain time interval, called the half-life of the radioactive element, half of the atoms will have decayed. This time interval may be determined by setting $N = N_0/2$ and $t = t_1/2$ with the result

$$\lambda t_1 = \ln 2$$

$$t_1 = \frac{0.693}{\lambda}$$

The mean lifetime is defined as

$$t_m = \frac{t_1}{\ln 2} = \frac{1}{\lambda}$$

and is just the reciprocal of the decay constant.

**Activity of a Radioactive Source**

The activity of a radioactive source is normally expressed as the number of disintegrations per unit time. A commonly used unit is the curie and is equal to $3.7 \times 10^{10}$ disintegrations per second. The curie was originally based on the rate of decay of a gram of radium. More convenient submultiples of the curie are the milli-curie ($1 \text{ mCi} = 10^{-3}$ curie) and the microcurie ($1 \mu\text{Ci} = 10^{-6}$ curie).

**Alpha Particle Energies**

The energies of known alpha particle emitters range from $8.9$ MeV for $^{212}\text{Po}$ to $4.1$ MeV for $^{232}\text{Th}$. Alpha particles may be detected and their energies determined very accurately with silicon surface barrier detectors.
Semiconductor Radiation Detectors

Conduction Particles

Charged particles or photons are detected in semiconductor detectors by the production of electron-hole pairs as the incident particle passes through the material. Both the electrons and holes may be collected and constitute a current. For a silicon detector an electron-hole pair is produced for every 3.5 eV of energy deposited in the detector or about $1.5 \times 10^6$ pairs for a 5 MeV incident particle.

Semiconductor detectors are just what their name implies; they are semiconductors and hence have a large number of charge carriers present in the crystal lattice—not as many as conductors but considerably more than insulators. In silicon at room temperature there are about $10^{10}$ pairs per cubic centimeter. This represents a considerable number of conduction particles and is far too large for Si to be used as a radiation detector. The reason Si has so many pairs of conducting particles is because the band gap between the valence and conduction bands is only 1.2 volts and at room temperature lattice vibrations sometimes produce electron hole pairs by breaking the covalent bonds between electrons and exciting them into the conduction band. In fact, this is the primary difference between insulators and semiconductors; the band gap is larger for insulators. A band structure diagram for silicon is shown in Figure 1.

![Band structure diagram for Si semiconductor.](image)

n-Type and p-Type Semiconductor Materials

It is possible to reduce the conductivity of silicon to a very low level and make it useful as a radiation detector. This is done by substituting another atom in the silicon lattice with one more valence electron than silicon. For example, silicon has four valence electrons and phosphorus has five which, after all covalent bonding is complete, leaves one electron which is weakly bound to the phosphorus atom and may be considered to be free.
Every phosphorus atom adds one free electron in the lattice and in addition reduces the number of holes present since the greater the number of electrons, the greater the probability that an electron will fall from the conduction band back into a hole in the valence band and hence reduce the number of holes. Thus, the intrinsic silicon which contained the same number of conduction holes and electrons has been changed by phosphorus doping to a material with a large number of conduction electrons and few holes. Therefore, the conduction of electric current will be primarily by the electrons. Since electrons have a negative charge the phosphorus-doped silicon is called an n-type semiconductor material.

It is also possible to produce a p-type material by doping silicon with a material which has one less valence electron than silicon. Boron only has three valence electrons. The electronic structure of boron is: \(1s^2\ 2s^2\ 2p^1\). Boron-doped silicon is a p-type material and current is conducted primarily by the motion of holes.

p-n Junction Diode

A detector is made by producing a p-n junction in a single piece of silicon. Figure 2(a) shows a single crystal doped with phosphorus on the right and boron on the left. The phosphorus-doped side has excess electron charge carriers and the boron-doped side has excess hole charge carriers. Figure 2(b) shows the same crystal with an electric field applied across the crystal. The free holes in the p-type material are driven to the left while the free electrons in the n-type material are driven to the right leaving a layer in the center of the crystal which is depleted of almost all charge carriers. This is called the depleted layer which acts as an insulator. This depleted layer has the characteristics required for a detector. An incident ionizing particle will leave a path of electron-hole pairs which will be swept out by the electric field and the charge collected will pass into an external circuit.

A silicon p-n junction detector is shown in Figure 2(c). The n-type material is made wafer thin so that the incident ionizing particle will lose only a minimum amount of energy before reaching the depleted layer. Typically, these layers of n-type material will be \(0.1\mu\) thick \((1\mu = 10^{-6} \text{ m})\).

Silicon n-p junction detectors have been made with surface areas up to \(1 \text{ cm}^2\) and sensitive thicknesses or depleted layers 1 mm thick. This thickness will completely stop a 10 MeV proton or a 40 MeV alpha particle.
a) p-n junction with zero applied bias.

b) p-n junction with an applied reverse bias. The arrow shows the direction of the electric field and the direction of motion of a positive charge.

c) silicon p-n junction radiation detectors

Figure 2
Experiment 1

The Use of Solid State Charged Particle Detectors for Alpha Particle Studies

Objective

To study the application of silicon surface barrier detectors to alpha-spectroscopy, to construct an energy calibration with a single alpha source and a pulse generator, to determine the absolute energy of an unknown alpha source.

References


Introduction

In this experiment we will outline the use of silicon surface barrier detectors for alpha spectroscopy. However, the techniques and methods used in the experiment will be applicable to many other experiments that will be done in the manual.

Surface barrier detectors for charged particle spectroscopy have wide application in physics and nuclear chemistry. These detectors can be constructed with active detecting areas as small as 1 mm² or as large as 25 cm². They are 100% efficient for alphas over their active area. Resolutions as low as 8 keV have been obtained for 5 MeV alpha particles. Perhaps the most impressive feature of the detectors is that they can be used for virtually any charged particle and their dynamic range is quite impressive. These have been used in experiments for 200 electron volt recoiling electrons and for 200 MeV heavy ion studies. For example, Figure 1 shows a pulse height spectrum of 120 keV scattered electrons from an accelerator.
Electron spectra for 120 KeV electrons scattered by an aluminum target at 40, 60, and 120 degrees.

On the other hand, Figure 2 shows a spontaneous fission fragment spectra from $^{252}$Cf. In this case, the upper energy fission group
is 110 MeV. There are many applications in nuclear chemistry in which charged particles are measured in order to establish some parameter in a chemical system. Surface barrier solid state radiation detectors do an excellent job in this capacity.

**Equipment**

1. **Radioactive Sources** as follows:
   a) 0.1 μCi $^{241}$Am
   b) 0.1 μCi $^{231}$Pa
   c) 0.5 μCi $^{230}$Th
   d) unknown alpha source

2. **Solid State Surface Barrier Detector** with the following parameters: resolution (30 keV), area (50 mm$^2$), depletion depth (100 microns)

3. **Bias Power Supply** for the Detector

4. **High Resolution Pre-amplifier**

5. **Spectroscopy Amplifier**

6. **Simple Vacuum Chamber and Pump**

7. **Nuclear Pulse Generator**

8. **Multichannel Analyzer** (~400 channels)

9. **Biased Amplifier** (optional; some multichannel analyzers have biasing capabilities built in)

10. **Oscilloscope**

**Procedure**

1. Set up the electronics as shown in Figure 3. Place the $^{241}$Am source in the vacuum chamber and pump down. Adjust the bias voltage to its recommended value. Set the gain of the amplifier so that the strong peak from $^{241}$Am falls about mid-scale in the multichannel. Figure 4 shows a spectrum of $^{241}$Am which indicates the two alpha groups that are seen if the multichannel has enough channel capacity. If the 5.48 MeV alpha is placed at mid-scale of a 400-channel analyzer, the two groups will only be separated by 1.5 channels and, hence, it is impossible to show the separation seen in Figure 4. Nevertheless, for this experiment it is not necessary to show the separation.

2. Accumulate a spectrum for the $^{241}$Am source for a period of time long enough to obtain about 1000 counts in the peak channel. Readout the multichannel and plot just the alpha peak as in Figure 4. Call the 5.48 MeV peak position channel C$_0$. 
Figure 3
Electronics for alpha spectroscopy with biased amplifier.

Figure 4
Spectrum of $^{241}\text{Am}$ taken with a surface barrier detector
3. Turn on the nuclear pulse generator and set its pulse height dial to 548/1000. Most pulse generators have pulse height dials that are ten-turn potentiometers. Each turn contains 100 divisions; and, hence, full scale on the dial represents 1000 divisions. It is thus convenient to express the dial setting as the ratio of the dial setting to the full scale setting. Observe where the peak is being stored in the multichannel. Adjust the normalize dial until the pulser peak falls exactly in channel C0. The pulse generator is now calibrated so that full scale on the pulse height dial corresponds to 10 MeV. Therefore, to generate a 6.0 MeV pulse, it is only necessary to set the pulse height dial to 600/1000, etc.

4. Erase the multichannel and store pulser pulses for about 20 seconds at a pulse height reading of 100/1000. Repeat for the other values that are shown in Table I. Readout the multichannel and fill in Table I. Turn off the pulse generator, but do not disconnect (disconnecting the connector may change the impedance and therefore the gain by a few channels).

Table I
Nuclear Pulse Generator Calibration Data

<table>
<thead>
<tr>
<th>Accumulation Time (approx., sec)</th>
<th>Pulse Height Dial Setting</th>
<th>Equivalent Energy (MeV)</th>
<th>Channel Number of MCA Peak</th>
</tr>
</thead>
<tbody>
<tr>
<td>20</td>
<td>100/1000</td>
<td>1.0</td>
<td></td>
</tr>
<tr>
<td>20</td>
<td>200/1000</td>
<td>2.0</td>
<td></td>
</tr>
<tr>
<td>20</td>
<td>300/1000</td>
<td>3.0</td>
<td></td>
</tr>
<tr>
<td>20</td>
<td>400/1000</td>
<td>4.0</td>
<td></td>
</tr>
<tr>
<td>40</td>
<td>500/1000</td>
<td>5.0</td>
<td></td>
</tr>
<tr>
<td>20</td>
<td>600/1000</td>
<td>6.0</td>
<td></td>
</tr>
<tr>
<td>20</td>
<td>700/1000</td>
<td>7.0</td>
<td></td>
</tr>
</tbody>
</table>

5. Turn off the detector bias supply, let the vacuum chamber up to air, and replace the 241Am source with 231Pa. Pump back down, turn on the bias and accumulate a spectrum (see Figure 5) for a time period long enough to be able to determine the major
peak positions in the spectrum. Readout the multichannel and fill in the channel positions as indicated in Table II.

![Graph](image)

**Figure 5**

Pulse height spectrum of $^{231}\text{Pa}$.

<table>
<thead>
<tr>
<th>Nuclide</th>
<th>$E_\alpha$ (MeV) Theory</th>
<th>Channel Number</th>
<th>$E_\alpha$ Measured (MeV)</th>
</tr>
</thead>
<tbody>
<tr>
<td>$^{231}\text{Pa}$</td>
<td>4.72</td>
<td></td>
<td></td>
</tr>
<tr>
<td>$^{231}\text{Pa}$</td>
<td>4.94</td>
<td></td>
<td></td>
</tr>
<tr>
<td>$^{231}\text{Pa}$</td>
<td>5.01</td>
<td></td>
<td></td>
</tr>
<tr>
<td>$^{223}\text{Ra}, ^{237}\text{Th}$</td>
<td>5.71</td>
<td></td>
<td></td>
</tr>
<tr>
<td>$^{219}\text{Rn}$</td>
<td>6.80</td>
<td></td>
<td></td>
</tr>
<tr>
<td>$^{215}\text{Po}$</td>
<td>7.36</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>
6. Replace the $^{231}$Pa source with $^{230}$Th and accumulate a spectrum for a time period which is sufficient to show the pronounced peaks for the source. Figure 6 shows a typical spectrum from $^{230}$Th. From the readout, determine the channel numbers associated with these peaks.

![Graph showing $^{230}$Th sample spectrum](image)

**Figure 6**

Pulse height spectrum of $^{230}$Th with decay scheme.
7. Obtain an unknown alpha emitting source from the instructor, replace the $^{230}\text{Th}$ source with this unknown and accumulate a spectrum as in Procedure 6. Determine the peak channels. Figure 7 shows a spectrum of $^{234}\text{U}$ which might have been used as an unknown.

Figure 7
Pulse height spectrum of $^{234}\text{U}$ with decay scheme.
8. Optional (the use of the bias amplifier in alpha spectroscopy). Set up the electronics as shown in Figure 8. Place the $^{231}$Pa source in the vacuum chamber, pump down, and turn on the detector bias voltage. Turn on the pulse generator and set its pulse height dial to 450/1000. Set the gain of the bias amplifier at 1 and the bias level control on the bias amplifier at 0/1000. Turn on the multichannel and visually observe the position of the pulser peak. It should be about mid-scale as it was in Procedure 3. Now, raise the bias level on the amplifier while visually observing the position of the peak on the multichannel. Continue raising this bias level until the pulser peak is being stored in the first few channels. We have now biased out all nuclear pulses below 4.5 MeV. Set the pulse generator for an 8 MeV output pulse (800/1000) and observe where the pulse is being stored in the last few channels of the analyzer. The multichannel is now calibrated from 4.5 MeV to 8.0 MeV.

![Figure 8](image)

Electronics for alpha spectroscopy with biased amplifier

9. Erase the multichannel and store the 8 MeV pulse for about 20 seconds. Set the pulser at 7.5 MeV and store for 20 seconds. Continue for the other values in Table III. Readout the analyzer and fill in the values. Note, the peak at 700/1000 was stored for 40 seconds rather than 20 seconds. The reason for this is so that it can be recognized on the readout. In other words, if you get mixed up in which peak is which, the 7 MeV point will help you since it has twice the counts of the other peaks.

10. Turn off the pulse generator, erase the multichannel and store a $^{231}$Pa spectrum for a long enough period to obtain good statistics in the pronounced peaks listed in Table II. Readout the multichannel.
Table III
Calibration with a Nuclear Pulse Generator and a Bias Amplifier

<table>
<thead>
<tr>
<th>Accumulate Time (approx., sec)</th>
<th>Pulse-Height Dial Setting</th>
<th>Equivalent Energy (MeV)</th>
<th>Channel Number of Peak</th>
</tr>
</thead>
<tbody>
<tr>
<td>20</td>
<td>800/1000</td>
<td>8.0</td>
<td></td>
</tr>
<tr>
<td>20</td>
<td>750/1000</td>
<td>7.5</td>
<td></td>
</tr>
<tr>
<td>40</td>
<td>700/1000</td>
<td>7.0</td>
<td></td>
</tr>
<tr>
<td>20</td>
<td>650/1000</td>
<td>6.5</td>
<td></td>
</tr>
<tr>
<td>20</td>
<td>600/1000</td>
<td>6.0</td>
<td></td>
</tr>
<tr>
<td>20</td>
<td>550/1000</td>
<td>5.5</td>
<td></td>
</tr>
<tr>
<td>40</td>
<td>500/1000</td>
<td>5.0</td>
<td></td>
</tr>
</tbody>
</table>

Data Reduction

Exercise (a)

From the data collected in Table I, plot a calibration curve of energy vs channel number. Figure 9 shows a typical calibration curve that was plotted for similar data. Determine the slope of the calibration curve. From the slope and the $^{241}$Am spectrum collected in Procedure 3 determine the resolution of the alpha group. Resolution is given by:

$$R = (\text{FWHM}) \times (\text{Slope of Calibration Curve})$$

where the FWHM is the full width at half maximum of the alpha peak. In other words, it is the width in channels of the peak at half of its height.

Exercise (b)

From the calibration curve and the readout of the $^{231}$Pa spectrum in Procedure 5, fill in the rest of the data in Table II.
How do your numbers compare with the accepted values in column 2 for this source? Determine the resolution of each of the groups in Table II.

Exercise (c)

Determine the energy and resolution of the unknown alpha source in Procedure 7 from your calibration curve.

(The rest of the exercises go with the optional portions of the experiment Procedures 7 through 10.)

Exercise (d)

Plot a calibration curve for the data collected in Table III. Determine the slope of the calibration curve. Note that the slope is quite different with this data as compared to that which was obtained in Table II. Figure 10 shows a typical plot for Table III. Note that in comparing Figures 9 and 10, the slope changed from 25 keV/channel to 9.2 keV/channel. The bias amplifier, therefore, allows one to look with better detail at a given spectrum.

Exercise (e)

From the calibration curve in Exercise (c) determine the energies and resolution of the pronounced peaks in the $^{231}\text{Pa}$ spectrum collected in Procedure 10.
What are nuclear isomers?

1.2 Define a curie.

1.3 An alpha particle disintegration is represented by a nuclear reaction equation of the type,

\[ A_{ZM1} \rightarrow A-4_{Z-2M2} + ^{4}_{2}\text{He} + Q_{\alpha} \]

where \( A_{ZM1} \) is the parent nucleus,

\( A-4_{Z-2M2} \) is the daughter nucleus,

and \( Q_{\alpha} \) is the disintegration energy and represents the total energy released in the process.
Evaluate the disintegration energy, $Q_0$, in terms of the kinetic energy of the alpha particle, $E_0$, using the principles of conservation of energy and momentum. Assume that the parent nucleus is at rest initially.

1.4 What are nuclear isobars?

1.5 Determine the number of alpha particles per second emitted by 5 mg of radium and express this number in millicuries.

1.6 Determine the amount of $^{210}$Po necessary to provide a 3 mCi source of alpha particles.

Computer Programs

The program GAUSS-6 can be used to find the centroids of all the peaks, and for the two calibration curves the slope can be found from LINEAR-6. Both of these programs are listed in the Appendix.

Additional References


Experiment 2

The Study of K and L Shell Binding Energies

Objective

To familiarize the student with the use of solid state surface barrier detectors for conversion electron spectroscopy, to measure conversion electron spectra and thus determine the K and L shell binding energies of $^{207}$Pb and $^{113}$In.

References


4. ORTEC Instruction Manual for Surface Barrier Detectors and the bibliography at the end of the manual which contains 157 references, available from ORTEC, Inc., 100 Midland Road, Oak Ridge, TN 37830.


Introduction

Many radioactive isotopes that are used in chemical and medical research decay by a process called internal conversion. In the internal conversion process it is possible for an excited nucleus to give its energy of excitation directly to one of the closely orbiting electrons. Since nuclear energies are in general higher than, for example, K-shell binding energies, the result is that the K electron is knocked out of the atom. If the conversion process occurs with an L-shell electron, then it is removed from the atom. In any case, internal conversion results in X rays and conversion electrons. If the energy of excitation of the nucleus is $E_x$, then the energy of the K-shell conversion electron is given by

$$E_e = E_x - K_{ab}$$

(1)
Where

\[ E_a = \text{energy of the conversion electron and} \]
\[ K_{ab} = \text{the binding energy of the K electron.} \]

The values of \( K_{ab} \) are listed in the back of this manual (Critical Absorption and Emission Energies).

If the conversion process occurs with an L electron, then the energy of the conversion electron is given by

\[ E_e = E_x - L_{ab} \quad (2) \]

For many nuclei the quantity \( E_x \) is well known; and, hence, Equations (1) and (2) can be used to determine the K- and L-shell binding energies of the daughter nucleus. If \( E_x \) is not known, then it can usually be determined by measuring the gamma-ray energy associated with the decay. For example, \(^{137}\text{Cs}\) decays by beta emission to the 662 keV level in \(^{137}\text{Ba}\), which then decays by gamma-ray emission or internal conversion. Hence, for a \(^{137}\text{Cs}\) source, one would see 662 keV gammas and K-internal conversion electrons whose energy are 662 keV minus \( K_{ab} \) for \(^{137}\text{Ba}\) (662 keV-36 keV=626 keV).

Radioactive sources for internal conversion measurements should be specially prepared for this application. The source should be thin and covered with a thin piece of Mylar.

Equipment

1. Radioactive Sources for Internal Conversion Measurements as follows:
   a) 5 \( \mu \text{Ci} \) \(^{137}\text{Cs}\)
   b) 5 \( \mu \text{Ci} \) \(^{207}\text{Bi}\)
   c) 10 \( \mu \text{Ci} \) \(^{113}\text{Sn}\)

2. Surface Barrier Detector: thickness (1000 microns), area (50 \( \text{mm}^2 \)), resolution (15 KeV for betas)

3. High Resolution Pre-amplifier

4. Spectroscopy Amplifier

5. Simple Vacuum Can in Which to Make the Measurements

6. Solid State Detector Bias Supply
8. Multichannel Analyzer (~400 channels)
9. Oscilloscope

Procedure

1. Set up the electronics as shown in Figure 1. Place the $^{137}$Cs source in the vacuum can and pump the system down until the mechanical pump is quiet. Adjust the bias voltage to the value recommended for the detector. Set the gain of the amplifier so that the output pulses from the $^{137}$Cs conversion line fall approximately mid-scale on the multichannel analyzer. (See Figure 2.) Accumulate a spectrum for a time period long enough to determine the centroid of the 624 keV group. Call this channel $C_0$.

2. Turn on the pulse generator and adjust the pulse height dial to 624/1000. This corresponds to 6.24 turns. Most commercial pulse generators have pulse height dials that are ten turn potentiometers. Adjust the normalize dial on the pulse generator until the pulser peak is being stored in the multichannel analyzer at exactly channel $C_0$. The pulser is now calibrated for 1000 keV full scale. Clear the multichannel and store pulser peaks for about 20 seconds each for the pulse height values shown in Table I. Readout the multichannel analyzer and record the channel positions for the indicated groups. The system is now calibrated.
Figure 2
Conversion electron spectrum from a $^{137}$Cs source.

Table I
Pulse Generator Calibration

<table>
<thead>
<tr>
<th>Pulse Height</th>
<th>Energy (keV)</th>
<th>Channel No.</th>
</tr>
</thead>
<tbody>
<tr>
<td>1000/1000</td>
<td>1000</td>
<td></td>
</tr>
<tr>
<td>800/1000</td>
<td>800</td>
<td></td>
</tr>
<tr>
<td>600/1000</td>
<td>600</td>
<td></td>
</tr>
<tr>
<td>400/1000</td>
<td>400</td>
<td></td>
</tr>
<tr>
<td>200/1000</td>
<td>200</td>
<td></td>
</tr>
</tbody>
</table>

3. Turn the bias voltage down to zero, remove the $^{137}$Cs source and replace it with a $^{207}$Bi source. Pump back down and adjust the bias voltage. Accumulate a spectrum for $^{207}$Bi for a time.
4. Remove the $^{207}\text{Bi}$ source and replace it with the $^{113}\text{Sn}$ source. Accumulate a spectrum for a long enough time to get good statistics in the L conversion electron group. Readout the multichannel. Figure 4 shows a spectrum that was taken for a $^{113}\text{Sn}$ source.

Data Reduction

Exercise (a)

From the information recorded in Table I, plot a calibration curve energy versus channel number. An example of such a plot is shown as the straight line in Figure 3. The ordinate is labeled as energy on the right hand axis. The slope of the calibration was 2.71 keV/ch for that amplifier gain setting. Determine the slope of your calibration curve.

Exercise (b)
Figure 4
Conversion electron spectra for a $^{113}\text{Sn}$ source.

Resolution = (FWHM) (Slope of calibration curve)
and the FWHM is the number of channels across the half height of
the peak.

Exercise (c)
From the calibration curve and the readouts taken for $^{207}\text{Bi}$
and $^{113}\text{Sn}$, fill in Table II.

Exercise (d)
From Equations (1) and (2) and the data in Table II, calculate
the K- and L-binding energies for $^{113}\text{In}$ and $^{207}\text{Pb}$. In doing this
for $^{113}\text{Sn}$, the assumption is $\Delta E = 0$. 

...
Table II

<table>
<thead>
<tr>
<th>Source</th>
<th>Conversion Line (keV)</th>
<th>Measured Energy (keV)</th>
</tr>
</thead>
<tbody>
<tr>
<td>$^{207}$Bi</td>
<td>1047</td>
<td></td>
</tr>
<tr>
<td>$^{207}$Bi</td>
<td>974</td>
<td></td>
</tr>
<tr>
<td>$^{207}$Bi</td>
<td>554</td>
<td></td>
</tr>
<tr>
<td>$^{207}$Bi</td>
<td>489</td>
<td></td>
</tr>
<tr>
<td>$^{113}$Sn</td>
<td>387</td>
<td></td>
</tr>
<tr>
<td>$^{113}$Sn</td>
<td>363</td>
<td></td>
</tr>
</tbody>
</table>

Exercise (e)

Calculate the difference in binding energy between the K and L shell for $^{113}$In assuming that you don't know the energy of excitation $E_x$. (Hint: write out Equations (1) and (2) and determine what has been experimentally determined.) Repeat for the two pairs of conversion electrons for $^{207}$Pb.

Exercise (f)

The ratio of conversions to the K and L shell for a given nucleus can be determined simply by summing under these various groups and then taking the ratio of the sums. Find these ratios for all the conversion spectra that were measured.

Post-Test

2.1 $^{137}$Cs decays by beta decay to the 0.662 MeV level of $^{137}$Ba which decays by gamma-ray emission or electron conversion. What are the kinetic energies of the K-and L-shell conversion electrons?

2.2 The 1062 keV gamma rays from $^{207}$Bi are incident upon a piece of uranium. What is the kinetic energy of the ejected K-shell electrons?
centroids of the peaks. LINEAR-6 can be used to obtain a least squares fit to the calibration data in Table I.

Additional References


Experiment 3

An Alpha Particle Time-of-Flight Experiment

Objective

To familiarize the student with "state-of-the-art" nuclear timing apparatus by measuring the flight time of an alpha particle over a given path length.

References


Introduction

In this manual there are several experiments that use silicon surface barrier detectors in a rather straightforward manner to study alpha particles, conversion electrons, or beta particles.

The recent advent of the thin transmission surface barrier detector makes other experiments, as well as the one described herein, possible. The transmission detector is a totally depleted device which will give an output pulse whose magnitude is proportional to the energy deposited in the detector by the charged particle being studied. For example, if a 7.36 MeV alpha particle goes through a transmission detector which is 26 microns thick, it will lose energy (ΔE) of approximately 3.61 MeV. Hence, the energy of the 7.36 MeV alpha will be reduced by the specific energy loss (dE/dx) in silicon of 3.61 MeV. The alpha particle which then emerges from the back of the ΔE detector will have an energy of 7.36 MeV minus 3.61 MeV or 3.75 MeV.
the thin transmission detector ($\Delta E$) to another detector which we will call the (E) detector. The physical arrangement for this experiment is shown in Figure 1.

![Figure 1](image)

**Figure 1**

Physical arrangement for alpha particle time-of-flight experiment.

**Equipment**

1. Vacuum Chamber (see Figure 1)
2. $\Delta E$ Surface Barrier Detector: area (50 mm$^2$), thickness (26 microns or less), resolution (20 keV), transmission mounted
3. E Surface Barrier Detector: area (100 mm$^2$), thickness (100 microns), resolution (25 keV)
4. Detector Bias Supplies for the Two Detectors
5. Nuclear Pulse Generator
6. Two Electronic Time Pick-off Units and Controls
7. Two High Resolution Pre-amps for the Detectors
8. Two Spectroscopy Amplifiers
9. 50 Ohm Delay Box (0-50 nsec)
10. Time-to-Pulse-Height Converter
11. Sum Delay Amplifier
12. Multichannel Analyzer (~400 channels)
Procedure

1. Set up the electronics as shown in Figure 2. The time pick-off unit derives a signal from the detector which is fed into the time-to-pulse-height converter (TPHC). The TPHC can be considered as an electronic clock. It produces an output pulse whose magnitude is proportional to the difference in time between its start and the stop pulse. Simply speaking, what is done in the experiment is: start the TPHC with the $\Delta E$ pulse and stop it with the $E$ detector pulse. The TPHC thus measures the flight time of the alpha particles between the two detectors. The output of the TPHC is amplified and fed into a multichannel analyzer. Before the alpha particle measurement is made, it is necessary to calibrate the TPHC. This is done with the nuclear pulse generator. Feed its output simultaneously into both the $\Delta E$ and $E$ time pick-off unit inputs. Set the delay shown in Figure 2 at 5 nanoseconds and determine the position of the peak in the multichannel analyzer (MCA). Repeat for the other values of delay shown in Table I and fill in the peak channel positions for each delay. After this measurement has been completed, set the delay back to zero.

<table>
<thead>
<tr>
<th>Delay n sec.</th>
<th>Peak Channel Position</th>
</tr>
</thead>
<tbody>
<tr>
<td>5</td>
<td></td>
</tr>
<tr>
<td>10</td>
<td></td>
</tr>
<tr>
<td>15</td>
<td></td>
</tr>
<tr>
<td>20</td>
<td></td>
</tr>
<tr>
<td>25</td>
<td></td>
</tr>
<tr>
<td>30</td>
<td></td>
</tr>
</tbody>
</table>

2. Place the $\Delta E$ and $E$ detectors about 2 cm apart (1.65 cm was used in the data that will be shown). Pump the system down and apply the recommended bias voltage for each detector. Accumulate a spectrum for a time period long enough to get
Figure 2
Electronics for alpha particle time-of-flight experiment.
3. Change the flight path between the two detectors to 10 cm and collect a spectrum in the MCA. Repeat for 15, 20, and 25 cm. Figure 3 shows a composite spectra that were taken for our example.

Figure 3
Time-to-pulse-height converter, output pulses for alpha particle time-of-flight experiment.

4. Return the flight path to 2 cm and collect a spectrum in the MCA from the sum amplifier. Since alpha particles lose part of their energy in the ΔE detector and the rest in the E detector, the sum of these two detectors should show the entire alpha particle spectrum. Figure 4 shows a typical sum spectrum for 231Pa. Figure 5 shows just the E spectrum. This is obtained by disconnecting the ΔE input.
Data Reduction

In this experiment the time of flight of the highest energy group in $^{231}$Pa is being measured. The other groups were biased out with a threshold discriminator on the time pick-off control. These 7.36 MeV alphas leave the $\Delta E$ detector with an energy of $\sqrt{3.75}$ MeV. An alpha of this energy has a reciprocal velocity of 75 nsec/meter. A flight path of 10 cm would give a time difference of 7.5 nsec which is quite easy to measure with the TPHC.

Exercise (a)

From the data collected in Table I plot a delay versus pulse height curve. This plot should be a straight line. The slope of the line in nsec/channel gives us a measure of our time resolution. Determine the slope from your data. The straight line
Figure 5
Pulse height spectrum from the (E) detector.

Exercise (b)

The time resolution of a peak is determined by multiplying the slope of the calibration curve times the FWHM of the peak. Determine the time resolution for the peaks in Table I.
Exercise (c)

The first alpha time-of-flight path length was 2 cm, the second was made for 10 cm. The path difference (\(\Delta d\)) between the two runs was therefore 8 cm. Determine the difference in the channel position of the peak for these two runs. Call this number \(\Delta C\). Obtain the corresponding \(\Delta t\) by the following:

1) \(\Delta t = (\Delta C)\) (slope of the calibration curve)

Determine the reciprocal velocity of the alpha group as follows:

2) \(\frac{1}{v} = \frac{\Delta t}{\Delta d}\)

The accepted value for the group was 75 nsec/m. Determine this value for the other path length differences used in the experiment. From all of the data taken, calculate an average reciprocal velocity.

Post-Test

3.1 Derive an expression for the flight time of an alpha particle in nsec in terms of its kinetic energy and distance of travel.

Computer Programs

GAUSS-6 can be used to find the centroids for the time calibration of the multichannel. LINEAR-6 can be used to fit the time calibration data and accurately establish the slope of the curve which is very important for this experiment.

Additional References


ANSWERS TO QUESTIONS AND PROBLEMS

Pre-Test Answers

1.1  The force is given by the familiar Coulomb's law for point charges,

\[ F = \frac{1}{4\pi \varepsilon_0} \frac{q_1 q_2}{r^2} \]

where \( q_1 \) and \( q_2 \) are the electronic charges, \( r \) is the distance separating the two electrons and \( \varepsilon_0 \) is the permittivity of free space and has the value

\[ \varepsilon_0 = 8.854 \times 10^{-12} \text{ coul}^2/\text{n-m}^2 \]

\[ F = \frac{1}{4\pi (8.854 \times 10^{-12} \text{ coul}^2/\text{n-m}^2)} \frac{(1.6 \times 10^{-19} \text{ coul})^2}{(0.05\text{m})^2} \]

\[ = 9.2 \times 10^{-36} \text{ n} \]

1.2  The nuclear coulomb barrier height is defined as the point where the coulomb barrier reaches a maximum value. This occurs at the nuclear surface of the atom of copper. The maximum height of the barrier is given by

\[ B = \frac{(Ze)(ze)}{R} \]

where \( Ze \) is the nuclear charge of the copper atom, \( ze \) is the incident proton charge, and \( R \) is the nuclear radius of copper.

\[ R = 1.2 \times A^{1/3} \text{ fermis} \]

\[ = 1.2 \times (65)^{1/3} \]

\[ = 4.82 \text{ fermis} \]

Multiplying both the numerator and denominator by Planck's constant divided by \( 2\pi \) and speed of light, \( (\hbar c) \), one gets

\[ B = \frac{Ze \cdot \hbar c}{R} \frac{\varepsilon_0 \cdot \hbar c}{\hbar c} \]

\[ = \frac{Ze \cdot \varepsilon_0}{\hbar c} \]
substituting

\[ e^2 \frac{\hbar}{c} = \text{the fine structure constant}, \alpha = \frac{1}{137} \]

and

\[ \hbar c = 197.32 \text{ MeV} - \text{fermis} \]

\[ B = \frac{(29)(1)}{(4.82 \text{ fermis})} \frac{1}{137} (197.32 \text{ MeV} - \text{fermis}) \]

\[ B = 8.76 \text{ MeV} \]

2.1 An electron volt is the kinetic energy acquired by an electron which is subjected to a potential difference of 1.0 volt.

2.2 keV = 10^3 eV

MeV = 10^6 eV

GeV = 10^9 eV

Post-Test Answers

1.1 Different energy states of the same nucleus. An isomeric state differs from an ordinary excited state of a nucleus in that it lasts for a measurable time.

1.2 A curie is a rate of decay and is equal to 3.7 \times 10^{10} disintegrations per second. It was originally based on the rate of decay of a gram of radium.

1.3 By the conservation of momentum, if an alpha particle is emitted of mass M and velocity V, the residual atom of mass M_2 will recoil with a velocity V_2.

\[ MV = M_2V_2 \]

Q_\alpha is the total energy released and equals

\[ Q_\alpha = \frac{1}{2}M_2(V_2)^2 + \frac{1}{2}MV_2^2 \]

where \( \frac{1}{2}MV_2^2 \) is the kinetic energy of the \( \alpha \) particle, E_\alpha. Eliminating V_2 from the above equations and substituting E_\alpha for the \( \alpha \) particle energy gives
To a very close approximation we can replace the ratio of the masses by the ratio of mass numbers

\[ \frac{M_1}{M_2} = \frac{4}{A - 4} \]

and the disintegration energy can be written

\[ Q_\alpha = E_\alpha \cdot \frac{A}{A - 4} \]

1.4 Nuclei which have the same mass number, A, but different atomic numbers, Z, are called isobars. Isobars are commonly produced in beta decay.

1.5 1 gm of radium produces $3.7 \times 10^{10}$ alpha particles per second and has an activity of 1 curie by definition.

\[ \frac{5 \text{ mg}}{1000 \text{ mg}} = \frac{N_\alpha}{3.7 \times 10^{10} \text{ disintegrations per second}} \]

\[ N_\alpha = 1.85 \times 10^8 \text{ disintegrations per second} \]

If 1 gm of radium has an activity of 1 curie, 5 mg would have an activity of 5 millicuries.

1.6 A 3 mCi source has an activity of $3 \times 10^{-3} \times (3.7 \times 10^{-10})$ or $11.1 \times 10^7$ disintegrations per second. The number of atoms of a radioactive isotope, dN, that will decay in a small interval of time, dt, is equal to the number of atoms present times the decay constant of the material:

\[ -\frac{dN}{dt} = \lambda N \]

Rewriting,

\[ N = \frac{1}{\lambda} \frac{dN}{dt} \]

where dN/dt is the activity of the sample and is equal to

\[ \frac{dN}{dt} = 11.1 \times 10^7 \text{ disintegrations per second} \]

Now

\[ \frac{1}{\lambda} = \frac{T_\frac{1}{2}}{0.693} \]
The number of $^{210}$Po can be determined from the following expression:

$$m(gm) = \frac{N A}{N_A}$$

where $A$ is the atomic weight of $^{210}$Po and $N_A$ is Avogadro's number.

$$m = (1.92 \times 10^{14} \text{ atoms}) \frac{(210 \text{ gm at wt})}{(6.024 \times 10^{23} \text{ atoms})}$$

$$= 6.69 \times 10^{-8} \text{ gm}$$

or

$$m = 0.067 \mu g$$

2.1 $E_k = 661.6 \text{ keV} - K_{ab}$

$$= 661.6 \text{ keV} - 37.4 \text{ keV}$$

$$= 624.2 \text{ keV}$$

$E_L = 661.6 \text{ keV} - L_{ab}$

$$= 661.6 \text{ keV} - 5.6 \text{ keV}$$

$$= 656.0 \text{ keV}$$

2.2 $E_k = 1062 \text{ keV} - K_{ab}$

$$= 1062 \text{ keV} - 115.6 \text{ keV}$$

$$= 946 \text{ keV}$$

3.1 The kinetic energy of an alpha particle is given by:

$$E = \frac{1}{2}mv^2$$

Rewriting, one obtains
or

\[
\frac{d}{t} = \sqrt{\frac{2E}{m}}
\]

Multiplying both numerator and denominator by the speed of light, \(c\),

\[
t = \frac{d}{\sqrt{E}} \sqrt{\frac{m}{2}} \frac{c}{\sqrt{c}}
\]

\[
t = \frac{d}{\sqrt{E}} \left[ \left( \frac{mc^2}{2} \right)^{\frac{1}{2}} \frac{1}{c} \right]
\]

\(mc^2\) is the rest mass energy of an alpha particle and is equal to 3725 MeV.

\[
t = \frac{d}{\sqrt{E}} \left[ \frac{\sqrt{3725}}{\frac{1}{0.3 \text{ m/nsec}}} \right]
\]

\[
t = \frac{144 \times d}{\sqrt{E}}
\]

where \(t\) is in nsec, \(d\) is in m, and \(E\) is in MeV.
INTRODUCTION

Neutron activation analysis was begun in the late 1930's. Since then it has become a very powerful technique for both qualitative and quantitative elemental identification.

Basically, a sample is irradiated by neutrons and becomes radioactive. By measuring the beta\(^+\), gamma rays produced, and the half lives of the components of the sample, the elemental composition and their concentrations may be determined.

Neutron activation is normally produced by means of natural radioactive sources, research reactors or neutron generators.

It is about 10 times more sensitive than other techniques of analysis such as mass spectroscopy and is comparable in sensitivity to X-ray fluorescence for some elements. For this reason, activation analysis is used extensively in such fields as geology, medicine, chemistry, metallurgy, and the petroleum industry.

This module consists of three experiments which may be performed by the student. The first experiment explores the use of thermal neutrons from radioactive sources for activation analysis. The second experiment investigates the use of fast neutron fluxes from an accelerator or radioactive sources. Identification of an unknown by neutron activation techniques is the subject of the third experiment.

Following this introduction are the objectives for the module, a list of prerequisites which the student should have before attempting this module, and a theoretical section which discusses some of the basic concepts of radioactive decay.

OBJECTIVES

To familiarize the student with the techniques used in neutron activation analysis; to measure the flux from fast and slow neutron sources; and to determine the qualitative and quantitative aspects of an unknown sample by neutron activation analysis.
1. Characteristics of Electromagnetic Radiation:
   a) Relationship between wavelength, frequency, and velocity.
   b) Relationship between energy and frequency of radiation; Planck's constant.

1.1 If the emission spectrum of NaI has a maximum at 4100 angstroms, what frequency does this correspond to?

1.2 What is the energy of the radiation in electron volts corresponding to this maximum in the emission spectrum?

2. Electron Volt (eV), keV, MeV:
   2.1 How is the electron volt defined?
   2.2 How is the eV related to the keV, MeV, GeV?

3. Interaction of Electromagnetic Radiation with Matter:
   a) Photoelectric effect
   b) Compton effect
   c) Pair-production process

3.1 What is the maximum kinetic energy of an electron ejected from the surface of a metal if the work required to free the electron from the surface is 6 eV and ultraviolet light of wavelength 150 Å is used to illuminate the surface?

3.2 Would the 1013 keV gamma ray from $^{27}$Al have a very large probability of interacting in a NaI detector by means of the a) pair-production process?, b) photoelectric effect?, or c) Compton effect?

THEORY

Gamma-Ray Scintillation Detector

Gamma radiation is detected in the scintillation phosphor by means of three processes: the photoelectric effect, the Compton effect, and electron-positron pair production. The scintillation phosphor converts the energy deposited in the detector by the incident ionizing radiation into light emission. The emitted light is allowed to impinge upon a sensitive photocathode of
The most desirable characteristics of a luminescent material are 1) a higher density for greater absorption of gamma rays, 2) increased pulse height for detection of low energy interactions, and 3) short decay times for fast counting and fast timing applications. NaI is the most commonly used inorganic scintillation phosphor primarily because of its large light output per unit energy deposited. Because of its extensive use, calculated efficiencies of NaI crystals are readily available (see for example, "Calculated Efficiencies of NaI Crystals" by E. A. Wolicki, R. Jastrow, and F. Brooks, NRL Report 4833).

Rate of Radioactive Decay

The rate of decay of a radioactive isotope is a constant for a particular material. The number of atoms, dN, which will decay in a time interval, dt, is equal to the number of atoms, N, present at the time, t, times the decay constant for the material.

\[-dN = \lambda N dt\]

Integrating, one obtains

\[N = N_0 e^{-\lambda t}\]

where \(N_0\) is the number of atoms present at time \(t = 0\). The number of atoms present at any given time decreases exponentially if no new atoms are produced.

At the end of a certain time interval, called the half-life of the radioactive element, half of the atoms will have decayed. This time interval may be determined by setting \(N = N_0/2\) and \(t = t_{1/2}\) with the result

\[\lambda t_{1/2} = \ln 2\]

\[t_{1/2} = \frac{0.693}{\lambda}\]

The mean lifetime of the radioactive isotope is defined as

\[t_m = \frac{t_{1/2}}{\ln 2} = \frac{1}{\lambda}\]

and is just the reciprocal of the decay constant.

Activity of a Radioactive Source
of a gram of radium. More convenient submultiples of the curie are the millicurie (1 mCi = 10⁻³ curie) and the microcurie (1 μCi = 10⁻⁶ curie).
Experiment 1

The Study of Thermal Neutrons by Activation Techniques

Objective

To accurately measure the thermal flux of neutrons from an isotopic neutron source or $^{252}$Cf source; to study the parameters associated with the buildup and decay of radioactivity; to measure the thermal neutron cross section of an isotope.

References


Introduction

In this experiment we are going to study a subject which very broadly fits the name activation analysis. If a sample is introduced into a region where there is a thermal neutron flux (n/cm$^2$-sec), the sample could absorb neutrons and become radioactive. The most pronounced absorption process for thermal neutrons is the so-called $(n,\gamma)$ reaction. Essentially what happens to an atom $A_1$ is the following: it absorbs a thermal neutron and then finds itself quite unstable. The resultant radioactive nucleus promptly emits a gamma and is still radioactive. From a nuclear equation notation the following happens:

$$A_1 + n \rightarrow A_1^{**} \rightarrow A_1^* + \gamma$$  \hspace{1cm} (1)

When we remove our sample from the neutron flux, the radioactive atoms in the sample are of the type $A_1^*$. For most radioactive atoms produced by this method, the most probable mode of decay is by beta emission promptly followed by gamma emission. Hence, measurements made on $A_1^*$ can either be made with a GM counter for the betas, or a gamma scintillation counter. In this experiment we are going to use a gamma scintillation counter.
The yield from an activation is

\[ A_0 = \frac{\sigma m z a S}{W} \]  

where

- \( A_0 \) = initial disintegrations/second of the whole element in the sample when the irradiation stops, i.e., at \( T = 0 \).
- \( \sigma \) = isotopic cross section for the reaction (cm\(^2\)).
- \( m \) = mass of the target element (gms).
- \( Z \) = Avogadro's number = \( 6.02 \times 10^{23} \) molecules/mole.
- \( \phi \) = neutron flux (neuts/cm\(^2\)-sec).
- \( a \) = fraction of target isotope in the target element.
- \( W \) = atomic weight, and
- \( S \) = saturation factor = \( (1 - e^{-\lambda t}) \),

where

- \( \lambda \) = decay constant = \( 0.693/T_h \) and \( T_h \) is the half life of the isotope
- \( t \) = time of irradiation, same units as \( T_h \).

The activation relation requires that \( A_0 \), the initial disintegration rate of the activated sample, be obtained. The sample activity is decaying during the period of measurement, and hence \( A_0 \) must generally be computed from the measurements. Moreover, a waiting period is frequently necessary because of the presence of unwanted, short-lived activity. If a sample is irradiated and then carried to a detector, some of the activity was lost in this process. Let's call \( T = 0 \) the time that we terminate the irradiation, \( T_1 \) the transfer time, and \( T_2 \) the counting period plus \( T_1 \). \( T_2 \) is the counting time period plus \( T_1 \) since it really is the total clock time after \( T = 0 \). Therefore, \( T_2 = T_1 \) and counting time (same units of time). Then the number of disintegrations during the counting period is given by:

\[ N_d = N_0 (e^{-\lambda T_1} - e^{-\lambda T_2}) \]

where \( N_0 \) is the initial number of radioactive atoms at \( T = 0 \).
\[ N_d = \frac{(E-B)}{\varepsilon F_Y} \]  

(4)

where

\( \varepsilon \) is the measured peak efficiency of the detector and

\( F_Y \) is the fraction of gammas per decay. Values of \( F_Y \) can be obtained from Reference 4.

Now all of the quantities in Eqs (3) and (4) are known except \( N_0 \). Hence, \( N_0 \), the initial number of radioactive atoms is given by:

\[ N_0 = \frac{(E-B)}{\varepsilon F_Y (e^{-\lambda T_1} - e^{-\lambda T_2})} \]  

(5)

but

\[ A_0 = \lambda N_0 \]  

(6)

therefore, \( A_0 \), the initial number of disintegrations per second is experimentally determined and can be plugged back into Eq (2) as a known quantity.

Therefore, if we take a known sample and irradiate it for a given length of time and count for a known time, we can experimentally determine \( \phi \), the neutron flux associated with the source and irradiating facility.

**Equipment**

1. Slow Neutron Source and Irradiator Facility
2. NaI Detector and Photomultiplier Tube
3. High Voltage Power Supply
4. Pre-amplifier
5. Spectroscopy Amplifier
6. Multichannel Analyzer (≈200 channels)
7. Aluminum and Vanadium Samples to be Irradiated (≈0.2 gms each depending on flux)
8. Calibration Sources as follows: 3 mCi \(^{131}\text{Cs}\), 5 mCi \(^{54}\text{Cr}\), 5 mCi \(^{60}\text{Co}\)
9. Unknown Cross Section Sample
Procedure

1. Set up the electronics as shown in Figure 1. Adjust the high voltage to the value recommended by the manufacturer. Set the gain of the amplifier so that the calibration range will allow you to measure 2 MeV full scale. Make an energy versus channel number calibration with $^{60}\text{Co}$, $^{137}\text{Cs}$, and $^{51}\text{Cr}$.

![Figure 1](image)

Electronics for Gamma Scintillation Spectrometry

2. Place an aluminum sample in the irradiator and irradiate it for 5 minutes. Terminate the activation and place the sample in the counting position. Wait an additional 1 minute from termination and then count for 2 minutes. From our definitions in the introduction:

$$t = 5 \text{ minutes}$$
$$T_1 = 1 \text{ minute}$$
$$T_2 = 3 \text{ minutes}$$

Figure 2 shows a typical spectrum that was collected under conditions similar to those just outlined. The cross section for the $^{27}\text{Al}(n,\gamma)^{28}\text{Al}$ reaction is 0.210 barns.

3. Activate the sample of vanadium and count it, using the following parameters: $t = 5 \text{ minutes}$, $T_1 = 1 \text{ minute}$, $T_2 = 6 \text{ minutes}$. Read-out the multichannel analyzer. Note the counting time with the multichannel analyzer is 5 minutes since $T_2 = T_1 + \text{counting time}$. Figure 3 shows a vanadium spectrum. The cross-section of the $^{51}\text{V}(n,\gamma)^{52}\text{V}$ reaction is 5.0 barns.
Figure 2
Thermal neutron activation spectrum of $^{28}$Al.

Figure 3
Thermal neutron activation spectrum of $^{52}$V.
4. The instructor will supply you with a known mass of one of the elements listed in Table I. The object of this experiment is to find the thermal neutron cross-section for the sample. Check Table I and see if your calibration range is such that the sample can be counted (most of the samples in the table fall in this category). With the proper calibration range, activate the sample for approximately one of its half lives, wait one minute and store a spectrum for a long enough period of time to get reasonable statistics under the photopeak. Readout the multichannel.

Table I

<table>
<thead>
<tr>
<th>Element</th>
<th>Target Nuclide</th>
<th>Target Material</th>
<th>Product Nuclide</th>
<th>T_{1/2}</th>
<th>E_{γ}, MeV</th>
</tr>
</thead>
<tbody>
<tr>
<td>Aluminum</td>
<td>^{27}\text{Al}</td>
<td>Al, Al\textsubscript{2}O\textsubscript{3}</td>
<td>^{28}\text{Al}</td>
<td>2.30 m</td>
<td>1.78</td>
</tr>
<tr>
<td>Sodium</td>
<td>^{23}\text{Na}</td>
<td>Na\textsubscript{2}CO\textsubscript{3}</td>
<td>^{24}\text{Na}</td>
<td>15.0 h</td>
<td>2.75, 1.37</td>
</tr>
<tr>
<td>Vanadium</td>
<td>^{51}\text{V}</td>
<td>NH\textsubscript{4}VO\textsubscript{3}</td>
<td>^{52}\text{V}</td>
<td>3.77 m</td>
<td>1.43</td>
</tr>
<tr>
<td>Manganese</td>
<td>^{55}\text{Mn}</td>
<td>MnO\textsubscript{2}</td>
<td>^{56}\text{Mn}</td>
<td>2.58 h</td>
<td>0.845, 1.81, 2</td>
</tr>
<tr>
<td>Cobalt</td>
<td>^{59}\text{Co}</td>
<td>CoO</td>
<td>^{60}\text{Co}</td>
<td>10.5 m</td>
<td>0.059 *</td>
</tr>
<tr>
<td>Copper</td>
<td>^{63}\text{Cu}</td>
<td>CuO</td>
<td>^{64}\text{Cu}</td>
<td>12.9 h</td>
<td>0.511 *</td>
</tr>
<tr>
<td>Gallium</td>
<td>^{71}\text{Ga}</td>
<td>Ga\textsubscript{2}O\textsubscript{3}</td>
<td>^{72}\text{Ga}</td>
<td>14.3 h</td>
<td>0.63, 0.83</td>
</tr>
<tr>
<td>Germanium</td>
<td>^{74}\text{Ge}</td>
<td>Ge</td>
<td>^{75}\text{Ge}</td>
<td>82 m</td>
<td>0.26, 0.20</td>
</tr>
<tr>
<td>Arsenic</td>
<td>^{75}\text{As}</td>
<td>As\textsubscript{2}O\textsubscript{3}</td>
<td>^{76}\text{As}</td>
<td>26.5 h</td>
<td>0.56, 0.65, 1</td>
</tr>
<tr>
<td>Bromine</td>
<td>^{79}\text{Br}</td>
<td>NH\textsubscript{4}Br</td>
<td>^{80}\text{Br}</td>
<td>18 m</td>
<td>0.62, 0.51 *</td>
</tr>
<tr>
<td>Indium</td>
<td>^{115}\text{In}</td>
<td>In</td>
<td>^{116}\text{In}</td>
<td>54 m</td>
<td>0.40, 1.09, 1.27, 2.08</td>
</tr>
<tr>
<td>Tellurium</td>
<td>^{130}\text{Te}</td>
<td>Te</td>
<td>^{131}\text{Te}</td>
<td>25 m</td>
<td>0.15, 0.45</td>
</tr>
<tr>
<td>Iodine</td>
<td>^{127}\text{I}</td>
<td>NH\textsubscript{4}I</td>
<td>^{128}\text{I}</td>
<td>25 m</td>
<td>0.46</td>
</tr>
<tr>
<td>Lanthanum</td>
<td>^{139}\text{La}</td>
<td>La\textsubscript{2}O\textsubscript{3}</td>
<td>^{140}\text{La}</td>
<td>40.2 h</td>
<td>0.48, 1.59</td>
</tr>
<tr>
<td>Tungsten</td>
<td>^{186}\text{W}</td>
<td>W\textsubscript{O}\textsubscript{3}</td>
<td>^{187}\text{W}</td>
<td>24 h</td>
<td>0.480, 0.686, 0.134</td>
</tr>
<tr>
<td>Gold</td>
<td>^{197}\text{Au}</td>
<td>Au-Dowex-1</td>
<td>^{198}\text{Au}</td>
<td>64.8 h</td>
<td>0.411</td>
</tr>
</tbody>
</table>
Data Reduction

Exercise (a)

From the multichannel readout in Procedure 1, determine the energy of the $^{28}$Al gamma. Does it agree with the value in Table I? Determine (E-8) for the photopeak and use the formulation in the introduction to determine $N_d$, $N_0$, $A_0$, and $\phi$. Remember, in Eq (2) $A_0$ is in disintegrations per second.

Exercise (b)

Repeat Exercise (a) for the vanadium monitor. Calculate $\phi$ and all the other parameters found in (a). Calculate the average flux value from the aluminum and vanadium data.

Exercise (c)

For Procedure 4 the instructor has provided you with all of the information that you need in regard to the sample. The unknown to be calculated is the thermal neutron cross section. Use the average flux value calculated in (b). Calculate $\sigma$, the thermal cross section for this sample.

Post-Test

1.1 Determine the fraction of the original radioactive atoms that decayed during a 4½ minute counting period for an isotope which has a half-life of 1.63 minutes, if a 30 second trans time was required.

1.2 If the number that decayed in Problem 1.1 was $8.2 \times 10^{12}$, how many radioactive atoms were there initially and what was the initial number of disintegrations per second?

Computer Programs

GAUSS-6 can be used to find the centroids of all of the peaks. LINEAR-6 will provide a linear least squares fit to the calibration data. GAUSS-6 can be used to find the thermal neutron flux in Exercises (a) and (b).

Additional References


Experiment 2

The Study of Fast Neutron Activation Techniques

Objective

To accurately measure the fast neutron flux from an accelerator or an unmoderated isotopic neutron source of $^{252}$Cf; to study the parameters associated with the buildup and decay of radioactivity; to measure the fast neutron cross section of a known isotope.

References


Introduction

The equations developed in Experiment 1 are equally applicable to problems in fast neutron activation analysis; and, hence, we will assume that the student has read Experiment 1 before starting this one.

There are three common sources of fast neutrons (in our case, fast means energies greater than 1 MeV) that are available to colleges and universities. These are: low voltage accelerators, isotopic neutron sources, and microgram quantities of $^{252}$Cf.
The low voltage accelerator in this application can generate neutrons by two reactions; these are:

\[ T + d \rightarrow n + ^{4}\text{He} \ (Q_m = 17.6 \text{ MeV}) \quad (1) \]

and

\[ D + d \rightarrow n + ^{3}\text{He} \ (Q_m = 3.2 \text{ MeV}) \quad (2) \]

The first reaction, which is the one most used by accelerator groups, generates 14 MeV neutrons. A deuterium beam of approximately 1 milliamp is allowed to strike a Zirconium Tritide target at an incident energy of 150 keV. The results can give neutron yields as high as \( 1 \times 10^{10} \text{ n/s} \) total yield. The flux at the irradiating position for this yield would be \( \sim 1 \times 10^{9} \text{ n/cm}^2\text{-sec} \) which is the quantity that we will measure. In Equation (2) Experiment 1, this quantity is called \( \phi \), the flux in \( \text{n/cm}^2\text{-sec} \).

The second source of fast neutrons is the isotopic neutron source. This is the type that many colleges and universities use. Basically, the source is composed of an alpha emitting isotope which is mixed with beryllium and fabricated in a stainless steel cylinder. The most common of these sources are the so-called Pu-Be or Ra-Be types. The neutrons are produced by the following reaction:

\[ ^{9}\text{Be} + \alpha \rightarrow n + ^{12}\text{C} \ (Q_m = 5.704 \text{ MeV}) \quad (3) \]

The flux of neutrons produced by one of these sources is shown in Figure 1. A one curie source will generally produce about \( 1.5 \times 10^6 \text{ n/sec} \). Note, in Figure 1 there is a distribution of energies and not just a single energy. The average energy for a Pu-Be source is about 5 MeV; and, hence, if we use an unmoderated source of this type in the experiment, the average cross section measured will be for 5 MeV neutrons.

Another source of neutrons that has recently become available to colleges and universities is \(^{252}\text{Cf}\). \(^{252}\text{Cf}\) is a spontaneously fissioning nucleus and, hence, the spectrum that would be observed is a typical fission spectrum. Figure 2 shows this spectrum for an unshielded \(^{252}\text{Cf}\) source. A 1 gram source will produce \( 2.34 \times 10^{12} \text{ n/s} \) in the distribution as shown in Figure 2. The sources that are being given to universities are usually a few micrograms and their yield would be \( \sim 5 \times 10^{6} \text{ n/sec} \) which is roughly equivalent to a 3 Ci Pu-Be source.
Figure 1
Flux of fast neutrons produced by a Pu-Be isotopic neutron source.

Figure 2
Spectrum of fast neutrons from an unshielded $^{252}\text{Cf}$ source.
Equipment
1. Fast Neutron Source and Irradiation Facility
2. NaI Detector and Photomultiplier Tube
3. High Voltage Power Supply
4. Pre-amplifier
5. Spectroscopy Amplifier
6. Multichannel Analyzer (~200 channels)
7. 0.5 gm Samples of Phosphorus and Chromium
8. Unknown Cross Section Sample
9. Calibration Sources $^{60}$Co, $^{137}$Cs, and $^{51}$Cr.

Procedure
1. Set up the electronics as shown in Figure 3. Calibrate the scintillation spectrometer for a full scale range of 2 MeV.

![Figure 3](image)

Figure 3
Electronics for neutron activation analysis.

2. Place the Phosphorus sample in the position to be irradiated and activate it for 3 minutes. Wait for 1 minute and count for 4 minutes. Therefore, in Eq (3) Experiment 1, $t = 3$ minutes, $T_1 = 1$ minute, and $T_2 = T_1 + 4 = 5$ minutes. After the spectrum has been accumulated, readout the multichannel and clear.
3. Repeat Procedure (2) for the chromium sample with the following parameters: \( t' = 4 \) minutes, \( T_1 = 1 \) minute, \( T_2 = 5 \) minutes. After the spectrum has been accumulated, readout the multichannel clear.

4. Obtain the unknown sample from the instructor and decide on the irradiation and counting parameters. Irradiate the sample and store a spectrum of the resulting activity.

Data Reduction

Exercise (a)

The phosphorus reaction for the activation process is given by

\[
^{31}\text{P} + n \rightarrow ^{31}\text{Si}
\]

The cross section for the reaction is 100 mb. The resultant gamma ray from \(^{31}\text{Si}\) has an energy of 1.266 MeV. Determine the energy experimentally from the calibration curve. From the multichannel output determine \((E-\delta)\) for the photopeak and solve Eq (2) through Eq (5) in Experiment 1 for the value of \(\phi\).

Exercise (b)

The chromium reaction for the activation process is given by

\[
^{52}\text{Cr} + n \rightarrow ^{52}\text{V}
\]

The cross section for the reaction is 80 mb. The resultant gamma ray from \(^{52}\text{V}\) has an energy of 1.44 MeV. From the calibration curve, find the experimentally determined energy for this gamma. From the multichannel output determine \((E-\delta)\) for the photopeak and solve Eqs (2) through (5) Experiment 1 to obtain another value for \(\phi\).

Exercise (c)

The instructor will provide you with all of the information about the unknown except the fast neutron cross section. From the readout find \((E-\delta)\) for your unknown and solve Eqs (2) through (5) for \(\sigma\), the cross section for the reaction. Note, use the average value for \(\phi\), the flux found in exercises (a) and (b). Table I gives some useful reactions that can be studied in this exercise. The gamma energies from the final radioactive nucleus can be found in Ref. 5.
### Table I

14 MeV Neutron Activation Cross Sections

<table>
<thead>
<tr>
<th>Element</th>
<th>Reaction</th>
<th>Average cross section ((\sigma)) in mb</th>
<th>Half-life</th>
<th>Radiations emitted</th>
</tr>
</thead>
<tbody>
<tr>
<td>Magnesium</td>
<td>(^{24}\text{Mg}(n,p)^{24}\text{Na})</td>
<td>1.1</td>
<td>15.0 hr</td>
<td>(\beta-, \gamma)</td>
</tr>
<tr>
<td>Aluminum</td>
<td>(^{27}\text{Al}(n,a)^{24}\text{Na})</td>
<td>0.57</td>
<td>15.0 hr</td>
<td>(\beta-, \gamma)</td>
</tr>
<tr>
<td>Titanium</td>
<td>(^{46}\text{Ti}(n,p)^{46}\text{Sc})</td>
<td>9.0</td>
<td>85 day</td>
<td>(\beta-, \gamma)</td>
</tr>
<tr>
<td></td>
<td>(^{47}\text{Ti}(n,p)^{47}\text{Sc})</td>
<td>15</td>
<td>3.43 day</td>
<td>(\beta-, \gamma)</td>
</tr>
<tr>
<td></td>
<td>(^{48}\text{Ti}(n,p)^{48}\text{Sc})</td>
<td>0.25</td>
<td>1.83 day</td>
<td>(\beta-, \gamma)</td>
</tr>
<tr>
<td>Manganese</td>
<td>(^{55}\text{Mn}(n,2n)^{54}\text{Mn})</td>
<td>0.18</td>
<td>314 day</td>
<td>EC, (\gamma)</td>
</tr>
<tr>
<td>Iron</td>
<td>(^{54}\text{Fe}(n,p)^{54}\text{Mn})</td>
<td>65</td>
<td>314 day</td>
<td>EC, (\gamma)</td>
</tr>
<tr>
<td>Nickel</td>
<td>(^{58}\text{Ni}(n,p)^{58}\text{Co})</td>
<td>90</td>
<td>72.0 day</td>
<td>EC, (\beta^+, \gamma)</td>
</tr>
<tr>
<td>Copper</td>
<td>(^{63}\text{Cu}(n,a)^{60}\text{Co})</td>
<td>0.42</td>
<td>5.27 yr</td>
<td>(\beta-, \gamma)</td>
</tr>
<tr>
<td>Zinc</td>
<td>(^{64}\text{Zn}(n,p)^{64}\text{Cu})</td>
<td>25</td>
<td>12.82 hr</td>
<td>(\beta-, \beta^+, \gamma)</td>
</tr>
<tr>
<td>Molybdenum</td>
<td>(^{92}\text{Mo}(n,p)^{92}\text{Nb})</td>
<td>6.0</td>
<td>10.1 day</td>
<td>EC, (\gamma)</td>
</tr>
<tr>
<td>Sulfur</td>
<td>(^{32}\text{S}(n,p)^{32}\text{P})</td>
<td>65</td>
<td>14.3 day</td>
<td>(\beta^-)</td>
</tr>
<tr>
<td>Phosphorus</td>
<td>(^{31}\text{P}(n,p)^{31}\text{S})</td>
<td>100</td>
<td>2.62 sec</td>
<td>(\beta^+, \gamma)</td>
</tr>
<tr>
<td>Chromium</td>
<td>(^{52}\text{Cr}(n,p)^{52}\text{V})</td>
<td>80</td>
<td>3.76 min</td>
<td>(\beta-, \gamma)</td>
</tr>
</tbody>
</table>

**Computer Programs**

GAUSS-6 can be used to find the centroids of all peaks. LINEAR-6 can be used to fit the calibration data. FLUX-6 can be used to solve Eqs (2) through (5) and, hence, find the flux of the irradiating facility.

**Additional References**


Experiments 3

Identification of an Unknown by Neutron Activation Analysis

Objective

To determine the qualitative and quantitative aspects of an unknown sample by slow neutron activation analysis. This includes an identification of the elements present in the sample by residual gamma-ray activity and half-life and comparison with standards to determine the amount of that element which is present.

References


Introduction

In this experiment we are going to assume that the student has already performed Experiment 1, The Study of Thermal Neutrons by Activation Techniques. It will also be assumed that, the flux of thermal neutrons (n/cm²·sec) has been determined.

Identifications of an unknown by activation analysis is accomplished in the following manner. The sample is first irradiated for a short (10 min) period of time. It is then counted to determine if that short irradiation produced any measurable activity. If the answer is yes, then some estimate is quickly made in regard to the half-life of the isotope that is produced. For example, let's assume that the half-life of the activity was 10 minutes. Then if we made a one-minute count every 2 minutes, we would be able to see the photopeak dying away as a function of time. With
isotope which was produced. The other fingerprint that is used is the measured gamma energy. By first calibrating the spectrometer, we can rather accurately determine the gamma energy of the resultant radionuclide. There are rather exhaustive tabulations of isotopes listed in accordance to increasing gamma energy. Reference 6 at the beginning of this experiment is perhaps one of the best. In Appendix VI of this manual we have tabulated the relative sensitivities of sixty isotopes relative to the $^{27}$Al($n,\gamma$)$^{28}$Al reaction. In the tabulation we have listed the half-life, measured gamma energy, and the relative sensitivity for thermal neutron activation. At the end of this tabulation we have made up a convenient coded chart that indicates the sensitivities of the elements as they appear in the Periodic Chart.

It is, therefore, assumed that with the aid of these charts, references, a set of carefully measured gamma spectra, and half-lives, the student will be able to find his unknown and standardize it in terms of the actual weight in the sample. This can be done even though the unknown may be complex in nature.

Equipment

1. Source of Thermal Neutrons and Irradiation Facility
2. 3" x 3" NaI Detector and Photomultiplier
3. High-Voltage Power Supply
4. Scintillation Pre-amplifier
5. Spectroscopy Amplifier
6. Multichannel Analyzer (~200 channels)
7. Calibration Sources: $^{60}$Co, $^{137}$Cs, $^{51}$Cr
8. Unknown Samples (several identical samples of each unknown)

Procedure

1. Set up the Electronics as shown in Figure 1. Except in a few cases, a full scale calibration of 2.5 MeV for the multichannel will be sufficient. Calibrate with $^{60}$Co, $^{137}$Cs, and $^{51}$Cr so that $^{60}$Co is about in the middle of the multichannel analyzer.
2. Irradiate the first unknown for a period of 10 minutes, wait 1 minute, and then count for a period of 5 minutes. The chances are good that under these conditions you will at least see gammas from your sample. Depending upon the amount of activity that you produced, you may proceed to either Procedure 3 or 4.
3. Assume that there is not much activity produced. If this is the case, the only thing that you can do is irradiate longer and count longer. Try some combination of both.

4. Assume that there is plenty of activity produced. This means that either there is a lot of the element present that is being studied, or that the half-life is short. It could be some combination of both. At this point, try to establish the half-life of the residual isotope. This can be done, for example, by taking counts in 5-minute intervals. Figure 2, for example, shows four spectra that were collected for $^{51}$Ti at 5-minute intervals.

5. From the calibration curve, you can also determine the gamma energies of the isotope. Figure 3 shows a spectrum of a Tungstic acid sample. The reaction that produced this activity was $^{146}$W(n,$\gamma$)$^{147}$W. The decay scheme for $^{147}$W can be found in The Table of the Isotopes, Reference 4. Also, one may use Reference 6 or Appendix VI at the back of this manual to help identify an unknown.

6. Assuming that you get the right answer for your unknown the next problem comes in the quantitative assay. For the purpose of this discussion, let's assume that the unknown was vanadium (see Figure 4). The student then weights up a known monitor of vanadium. Irradiate it and count for reasonable statistics. Repeat this exact measurement with the vanadium unknown. Remember the laboratory instructor provided you with several identical samples of the unknown. Be sure to compare the standard to a vanadium unknown that has not already been irradiated.
Figure 2
Series of four pulse height spectra for $^{51}\text{Ti}$ showing the fall-off in activity in time.
Figure 3
Neutron activation spectrum of tungsten.

Figure 4
Pulse height spectrum of activated vanadium metal.
Data Reduction

Exercise (a)

From your calibration curve determine the gamma energies of the unknown. The half-life data, along with the gamma energies, should provide you with enough data to determine what isotope it is that you are measuring.

Exercise (b)

In your comparative data determine \((\gamma, \beta)\) for both the standard and the unknown. If all irradiation parameters were identical, the ratios of photopeak sums is the same as the ratios of the masses of the standard and the unknown.

Exercise (c)

The method used in Exercise (b) is called the relative method of comparison. It is also possible to determine the amount of an unknown isotope by the absolute method.

From Experiment 1

\[
\text{Eq (1)} \quad \lambda_0 = \frac{\sigma m z a s}{W}
\]

In this equation we know, or can experimentally determine, everything except \((m)\), the mass of the sample in grams. Experiment 1 carefully outlines how this can be done. Determine \((m)\) by this method.

Computer Programs

GAUSS-6 can be used to find the centroids of all of the peaks. LINEAR-6 will provide a linear least squares fit to the calibration data. GAUSS-6 can be used to find the thermal neutron flux in exercises (a) and (b).

Additional References


ANSWERS TO QUESTIONS AND PROBLEMS

Pre-Test Answers

1.1 $\lambda = 4100 \text{ Å} \times 10^{-8} \text{ cm/Å}$

$= 4100 \times 10^{-8} \text{ cm}$

$f = \frac{c}{\lambda}$

$= \frac{3 \times 10^{10} \text{ cm/sec}}{4100 \times 10^{-8} \text{ cm}}$

$= 7.3 \times 10^{14} \text{ sec}^{-1}$

1.2 $E = hf$

$= (6.625 \times 10^{-27} \text{ erg} \cdot \text{sec}) \times (7.3 \times 10^{14} \text{ sec}^{-1})$

$= 48.5 \times 10^{-13} \text{ erg} \times \frac{1 \text{ ev}}{1.602 \times 10^{-12} \text{ erg}}$

$= 30.3 \times 10^{-1} \text{ ev}$

$= 3 \text{ ev}$

2.1 An electron volt is the energy acquired by an electron in passing through a potential difference of 1 volt.

2.2 1 keV = 1000 ev

1 MeV = 10^6 ev

1 GeV = 10^9 ev

3.1 $\lambda = 150 \text{ Å}$

$= 150 \times 10^{-8} \text{ cm}$

$f = \frac{c}{\lambda}$

$= \frac{3 \times 10^{10} \text{ cm/sec}}{150 \times 10^{-8} \text{ cm}}$

$= 2 \times 10^{16} \text{ sec}^{-1}$
\[
E_{\text{max}} = hf - W \\
= (6.625 \times 10^{-27} \text{ erg sec}) (2 \times 10^{16} \text{ sec}^{-1}) - 6 \text{ eV} \\
= 13.25 \times 10^{-11} \text{ erg} - 6 \text{ eV} \\
= 76.7 \text{ eV}
\]

3.2 a) No, because 1022 keV of energy is required to produce the electron-positron pair.

b) Yes, with a Compton interaction approximately 20 times more likely than a photoelectric interaction.

**Post-Test Answers**

1.1 The decay constant for this isotope is
\[
\lambda = \frac{\ln 2}{t_\frac{1}{2}} \\
= \frac{0.693}{93 \text{ sec}} \\
= 0.00745 \text{ sec}
\]

The number of atoms which decayed is
\[
N_d = N_0 (e^{-\lambda T_1} - e^{-\lambda T_2})
\]

where \(N_0\) is the original number of radioactive atoms, \(T_1\) is the transfer time and \(T_2\) is the transfer time plus counting time.

\[
N_d = N_0 \left[e^{-(0.00745 \text{ sec}^{-1})(30 \text{ sec})} - e^{-(0.00745 \text{ sec}^{-1})(300 \text{ sec})}\right] \\
= N_0 (e^{-0.224} - e^{-2.24}) \\
= N_0 (0.6928)
\]

\[
\frac{N_d}{N_0} = 0.6928
\]
1.2 \( N_d = 8.2 \times 10^{12} \) disintegrations

\[
N_0 = \frac{N_d}{0.6928} = \frac{8.2 \times 10^{12}}{0.6928} = 1.18 \times 10^{12} \text{ atoms}
\]

\[
A_0 = \lambda N_0 = (0.00745 \text{ sec}^{-1})(1.18 \times 10^{12} \text{ disintegrations}) = 8.8 \times 10^9 \text{ disintegrations/sec}
\]
INTRODUCTION

Photon attenuation is very important especially for low energy photons. For example, a 10 keV photon has 100 times the probability of being absorbed as a 1000 keV photon.

This experiment involves the measurement of the linear absorption coefficient for low energy X rays. Therefore, we will be determining the number of events that are observed under various experimental conditions. There are unique problems associated with X-ray absorption measurements and this experiment will be concerned with the solutions of these problems. Ideally, the experiment should be performed in vacuums; however, for 8 keV X rays passing through a few centimeters of air, the corrections are small and may be neglected. For X rays below 8 keV the corrections become increasingly more important and attenuation experiments performed in this region should be done in a special vacuum chamber. As an example, a 2 keV X-ray beam would be attenuated about 80% by a piece of ordinary paper.

The X rays will be detected with a gas-filled proportional counter. It will be assumed that the student is already familiar with the use of proportional counters with X-ray sources.

OBJECTIVES

To study the attenuation of low energy X rays when they pass through thin foils; to determine the half value thickness for aluminum and nickel for the Kα X rays from Zn-65.

PREREQUISITES WITH PRE-TEST QUESTIONS AND PROBLEMS

1. Characteristics of Electromagnetic Radiation:
   a) Relationship between wavelength, frequency, and velocity.
   b) Relationship between energy and frequency of radiation, Planck's constant.

1.1 What is the frequency of the Kα1 X ray from Selenium which has an energy 11.321 keV?
1.2 What is the wavelength of this X ray?

2. Electron volt (eV), keV, MeV:
   2.1 How is an electron volt defined?
   2.2 How is the eV related to the multiples keV, MeV, GeV?

3. Interaction of Electromagnetic Radiation with Matter:
   a) Photoelectric effect
   b) Compton effect
   c) Pair production process

3.1 How is the mass attenuation coefficient related to the linear attenuation coefficient?

3.2 Derive the general relativistic expressions for the maximum forward momentum of a photoelectron ejected by a photon of energy hf and the corresponding recoil momentum of the residual atom. Neglect the binding energy between the electron and the atom and the kinetic energy of the recoiling atom in comparison to the energy of the incident photon.

THEORY

At energies below 20 keV, photons interact with matter primarily by the photoelectric process. Compton and pair production cross sections are for the most part unimportant in this energy range. Let us assume that a thin piece of metal is placed between the X-ray source and the detector.

From Lambert's law (Reference 1) the decrease in intensity of the radiation as it passes through the foil is given by:

\[ I = I_0 e^{-\mu x} \]

where:

- \( I \) = intensity after the absorber
- \( I_0 \) = intensity before the absorber
- \( \mu \) = total mass absorption coefficient (cm\(^2\)/gm)
- \( x \) = density thickness in gm/cm\(^2\)

The density thickness is the product of density in gm/cm\(^3\) times
The half value layer (HVL) is defined as the thickness of the absorbing foil which will reduce the original intensity to one-half of its initial value.

\[
\frac{I}{I_0} = e^{-\mu x}
\]

\[
\ln\left(\frac{I}{I_0}\right) = -\mu x
\]

if \( \frac{I}{I_0} = 0.5 \)

then \( x = \text{HVL} \)

therefore \( \ln(0.5) = -\mu(\text{HVL}) \)

and \( \text{HVL} = \frac{0.693}{\mu} \)

Experimentally the (HVL) is determined by inserting foils of various thicknesses between the source and the detector and observing the fall off in intensity. A plot on a log scale of intensity versus absorber thickness will give a straight line. The slope of the line is \(-\mu\) and the HVL can be determined from the above information.

REFERENCES


EQUIPMENT

Option A


2. Proportional Counter Pre-amplifier
3. High Resolution Spectroscopy Amplifier
4. Proportional Counter High Voltage Power Supply
5. 10 μCi $^{65}$Zn X-Ray Source, (10 μCi $^{57}$Co Source, Optional)
6. Multichannel Analyzer (100 channel or more)
7. Thin Foils of Nickel and Aluminum up to a T of 70 mg/cm$^2$

Option B (Items 1 through 7 plus . . .)
8. Single Channel Analyzer
9. Electronic Digital Scaler
10. Electronic Timer
11. Linear Gate
12. Delay Amplifier (1 to 5 µsec)

PROCEDURE

Option A

1. Set up the electronics as shown in Figure 1. Place the 10 μCi $^{65}$Zn source about 2 cm from the face of the proportional counter. Set the high voltage power supply to the value recommended by the manufacturer. Adjust the gain of the amplifier so that the K$_\alpha$ group from the $^{65}$Zn source is being stored midway in the multichannel analyzer.
2. Accumulate a spectrum for a period of time long enough to obtain 6000 counts under the K X-ray group. Remove the source and collect a background spectrum for the same length of time. Subtract the background counts which would fall under the K X-ray group.

Call the resulting value \((\Sigma-\beta)\). Divide by the time and thus determine \([I_0 = \frac{(\Sigma-\beta)}{t}]\). This term represents the number of counts per second that the detector observes without an absorbing filter.

3. Replace the source and insert a 5(mg/cm²) aluminum foil between the source and the detector. Determine \((\frac{(\Sigma-\beta)}{t})\) as in 2.

4. Repeat this same measurement for the thicknesses recorded in Table I. Fill in the data table.

Table I

<table>
<thead>
<tr>
<th>Absorber</th>
<th>Thickness mg/cm</th>
<th>((\Sigma-\beta)/t = I_0)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>5</td>
<td></td>
</tr>
<tr>
<td>2</td>
<td>10</td>
<td></td>
</tr>
<tr>
<td>3</td>
<td>15</td>
<td></td>
</tr>
<tr>
<td>4</td>
<td>20</td>
<td></td>
</tr>
<tr>
<td>5</td>
<td>25</td>
<td></td>
</tr>
<tr>
<td>6</td>
<td>30</td>
<td></td>
</tr>
<tr>
<td>7</td>
<td>35</td>
<td></td>
</tr>
<tr>
<td>8</td>
<td>45</td>
<td></td>
</tr>
<tr>
<td>9</td>
<td>60</td>
<td></td>
</tr>
</tbody>
</table>

5. Make a similar data table for nickel foils and repeat the measurements for the values in the table.
Nickel Filters

\[ \text{\^{65}Zn Source} \]

8.05 KeV X-Ray

Half Value Thickness: 15.7 mg/cm²

Mass Attenuation Coefficient: 44.1 cm²/gm

Figure 2

Attenuation Measurement for Nickel for 8.05 KeV X Rays from \( \text{\^{65}Zn} \)

Aluminum Filters

\[ \text{\^{65}Zn Source} \]

8.05 KeV X-Ray

Half Value Thickness: 14.0 mg/cm²

Mass Attenuation Coefficient: 49.5 cm²/gm

Figure 3

Attenuation Measurement for Aluminum for 8.05 KeV X Ray from \( \text{\^{65}Zn} \)
DATA REDUCTION

Exercise (a)

Using semi-log graph paper, plot I versus the absorber thickness. Figures 2 and 3 show similar plots for nickel and aluminum. Determine the HVL for these two metals.

Exercise (b) (Optional)

Remove the $^{65}$Zn source and place a 10 µCi $^{57}$Co source in the source position. Repeat the attenuation measurement for enough absorber thicknesses to establish a graph similar to Figures 2 and 3. Figure 4 shows a pulse height spectrum for $^{57}$Co that was measured with a proportional counter. Note that in this measurement there are two photon groups; and, hence, if they are treated separately, the HVL of both the 6.4 and the 14.4 keV groups can be determined.

![Proportional Counter X-Ray Spectrum from $^{57}$Co](image)

Figure 4

Proportional Counter Pulse Height Spectrum for $^{57}$Co
PROCEDURE

Option B

1. Set up the electronics as shown in Figure 5. Place the $^{65}$Zn source 2 cm from the face of the proportional counter. Set the high voltage at the value recommended by the proportional counter manufacturer. Adjust the gain of the amplifier so that the 8.04 keV line from $^{65}$Zn gives output pulses from the amplifier at about 4 volts. Set the delay amplifier at 2 μsec. Set the single channel analyzer in the differential mode of operation and adjust the E and ΔE dials so that the single channel analyzer brackets the output pulses from the amplifier.

X-RAY ABSORPTION MEASUREMENTS

2. With a scope look at the output pulses from the linear gate. The output pulses from the linear gate should look just like the pulses from the amplifier. The linear gate is a coincidence type circuit that will pass an input pulse from the amplifier into the multichannel, provided there is a coincident (ENABLE) pulse from the single channel analyzer. The (ENABLE) pulses really open the linear gate which in turn passes the amplifier pulses into the multichannel analyzer. Since the scaler is also recording the single channel analyzer output pulses, it is essentially integrating under the peak that is observed in the multichannel analyzer. Thus this circuitry allows one to
by-pass the time associated with reading out the multichannel and summing the peak in the spectrum.

3. Now that the system is adjusted, the scaler is used for the measurements. Set the timer for a time which is sufficient to give about 6000 counts in the scaler. Record the time. Remove the source and accumulate a background measurement for the same period of time. Determine \( (E - B)/t = I \) as in Option A.

4. Place the 5 mg/cm² Nickel foil between the source and the detector and run for the same length of time as 3. Determine I for this measurement. Continue for the foil thicknesses in Table I for both nickel and aluminum. Exercises (a) and (b) same as for Option A.

DISCUSSION

It is possible to produce X rays by fluorescence and then measure their attenuation as outlined above. Figure 6 shows one possible arrangement by which this can be done. The student would benefit by first reading Experiment 3 in Module 1 which is X-Ray Fluorescence with a Proportional Counter. The advantage of this technique is that any X-ray energy which is desired can be produced by the proper choice of the target element which is being fluoresced. The rest of this exercise is composed of filling in data Table I as in (a) and (b) above.

X-RAY FLUORESCENCE USING PROPORTIONAL COUNTER

![Diagram of X-ray fluorescence setup]

Figure 6

Absorption Measurements with Fluorescent X Rays
POST-TEST

1. What fraction of 847-keV gamma rays incident on a 3 cm thick sheet of Fe are attenuated if the linear attenuation coefficient is 0.511 cm\(^{-1}\)?

2. 6.4-keV K\(_x\) X rays from Fe are detected by a Lithium drifted silicon detector with a 10 mil (1 mil = 0.001") Be window positioned 10 cm from the Fe target. What is the correction for attenuation in the air and Be window if the linear attenuation coefficients are 0.023 cm\(^{-1}\) and 5.65 cm\(^{-1}\), respectively?

3. Would 30-keV X-ray photons have a larger probability of interacting by means of a) photoelectric effect, b) Compton effect, or c) pair production process and why?

ADDITIONAL REFERENCES


Pre-Test Answers

1.1 $E = hf$

\[
f = \frac{11,221 \text{ eV}}{6.625 \times 10^{-27} \text{ erg-sec}}
\]

\[
= (1693.7 \times 10^{27} \text{ eV/erg-sec}) \times (1.602 \times 10^{-12} \text{ erg/eV})
\]

\[
= 2.713 \times 10^{18} \text{ sec}^{-1}
\]

1.2 $\lambda = \frac{c}{f}$

\[
= \frac{3 \times 10^{10} \text{ cm/sec}}{2.713 \times 10^{18} \text{ sec}^{-1}}
\]

\[
= 1.106 \times 10^{-8} \text{ cm}
\]

\[
= 1.106 \AA
\]

2.1 The eV is the energy acquired by an electron in passing through a potential difference of 1 volt.

2.2

1 keV = 1000 eV

1 MeV = 10^6 eV

1 GeV = 10^9 eV

3.1 The mass attenuation coefficient $\mu/\rho (\text{cm}^2/\text{g})$ is the linear attenuation coefficient $\mu (\text{cm}^{-1})$ divided by the density of the material $\rho (\text{g/cm}^3)$. The mass attenuation coefficient is really more fundamental since it is independent of the actual density and physical state of the absorber.

3.2 By conservation of total relativistic energy

\[
E_\gamma + E_{e_i} + E_{a_i} = E_{e_f} + E_{a_f}
\]

But, $E_\gamma$ can be rewritten as

\[
E_\gamma = hf = \frac{hc}{\gamma} = p_\gamma c,
\]
If we neglect the recoil kinetic energy of the atom with respect to the energy of the incident photon, then we have:

\[ P_{\gamma} c + m_{e} c^2 = \sqrt{P_{e}^2 c^2 + m_{e}^2 c^4} + \sqrt{P_{a}^2 c^2 + m_{a}^2 c^4} \]

Solving for the maximum momentum of the photoelectron, we get:

\[ P_{e} = \sqrt{P_{\gamma}^2 + 2P_{\gamma} m_{e} c} \]

By conservation of momentum,

\[ P_{\gamma} = P_{e} - P_{a} \]

Hence,

\[ P_{a} = \sqrt{P_{\gamma}^2 + 2P_{\gamma} m_{e} c} - P_{\gamma} \]

Post-Test Answers

1. The fractional transmission is

\[ \frac{I}{I_0} = e^{-\mu x} \]

\[ = e^{-0.511 \text{ cm}^{-1}}(3 \text{ cm)} \]

\[ = 0.216 \]

The fraction that is attenuated is

\[ 1 - \frac{I}{I_0} = 0.784 \]
2. The fraction that was not attenuated by the air and Be window is given by:

\[ \frac{I}{I_0} = \left[ e^{-0.023 \, \text{cm}^{-1}}(10 \, \text{cm}) \right] \left[ e^{-5.65 \, \text{cm}^{-1}}(0.0254 \, \text{cm}) \right] \]

\[ = (0.793) (0.866) \]

\[ = 0.686 \]

Correction = \[ \frac{I_0}{I} \]

\[ = \frac{1}{0.686} \]

\[ = 1.46 \]

3. Because of the relatively low energy of X-ray photons (0-100 keV), the probability of interacting is larger for the photoelectric effect than by the Compton effect. Photoelectric interactions are possible only for small hf and large Z, i.e. \( g_{ph} \approx Z^4/(hf)^3 \). Compton collisions predominate in the entire region of intermediate hf (i.e. 100 keV < hf < 10,000 keV) and for all Z. Pair production is only important for large hf (>1022 keV) and large Z.
MODULE FIVE
ACCELERATOR EXPERIMENTS

A module utilizing low voltage accelerators in atomic and nuclear physics experiments.

INTRODUCTION

Before accelerators were used, natural radioactive sources and cosmic rays provided the only energetic sub-atomic particles for the study of nuclear properties. Since the early 1930's charged particle accelerators have been used extensively to advance our basic understanding of atomic and nuclear structure. They provide the experimenter with a vast number of different projectiles of controllable energy and intensity. The earliest accelerators could produce proton beams from less than 100 keV to 1 MeV in energy. The four basic types of accelerators are the Cockcroft-Walton, the linear accelerator, the cyclotron, and the Van de Graaff.

This module consists of two experiments. The first experiment involves the measurement of thin films by elastic ion-beam scattering. The second experiment studies X-ray production using a small accelerator.

Following this introduction are the objectives for the module and a list of desirable prerequisites for the student to have before attempting this module.

OBJECTIVES

The objectives of this module are to familiarize the student with the equipment and techniques used in experiments utilizing small accelerators. Specifically, the objectives are to measure, the thickness of thin films by elastic ion scattering and to study X-ray production by proton bombardment.

PREREQUISITES WITH PRE-TEST QUESTIONS AND PROBLEMS

1. Electron Volt (eV), keV, MeV, GeV:
   1.1 How is the eV related to the multiples keV, MeV, GeV?
   1.2 An eV is equivalent to how many ergs? Joules?

2. Relationship between Wavelength, Frequency, Velocity, and Energy of Electromagnetic Radiation:
2.1 What is the frequency and wavelength of the 11.92 keV \( K_a \) X-ray photon from Bromine?

3. Coulomb Force:

3.1 What is the coulombic force of attraction between the electron and the proton for the hydrogen atom in its ground state?

4. Magnetic Force on a Moving Charge:

4.1 What is the magnetic force on a 150 keV proton moving perpendicular to a uniform magnetic field of 6 kilogauss?

5. Rutherford Scattering Cross Section:

5.1 What is the expression for the Rutherford scattering differential cross section?

5.2 How does the Rutherford cross section vary as a function of the incident projectile charge? the target nuclear charge? the energy of the incident projectile?
Experiment 1
Thin Film Measurements by Elastic Ion Beam Scattering with a Low Voltage Accelerator
(A Laboratory Experiment & Senior Research Project)

Objective
To use a low voltage accelerator in the study of thin films by measuring the scattered ions from the sample.

References

Introduction
In this experiment we are going to bombard a thin target with protons or deuterons from a low voltage accelerator. The accelerator can be a Cockcroft-Walton or Van de Graaff type. The experimental write-up is intended for machines that have accelerating voltages less than 500 keV. Figure 1 shows a photograph of a typical 400 keV Van de Graaff, bending magnet and scattering chamber that is set up for educational experiments. Figure 2 shows a photograph of a bending magnet and scattering chamber. In general, the ion beams that are used for this application are quite low in intensity. Beam currents for this application are usually less than 50 namps. At these low voltages and beam currents, there is essentially no health physics problems generated at the target. However, a person who is
familiar with the accelerator and its operations should be involved with the experiments until the new users are familiar with the accelerator parameters.

Figure 3 shows an overall schematic of the accelerator and the associated electronics for this experiment. A scattering chamber of some sort has to be constructed or purchased for this experiment. Basically, the scattering chamber is a cylinder that mounts to the end of the beam tubing. It has in it the following: a mounting frame for the thin foil which is our target, a movable solid state surface barrier detector for measuring the elastically scattered ions, an isolated Faraday cup for measuring the number of protons that impinge on the target for a given measurement, and defining slits to adjust the size of the beam spot on the target. Figure 4 gives an artist's concept of a scattering chamber with the basic features which are necessary for this experiment.

In this experiment we will simply record the number of scattered particles that come from the target at an angle $\theta$ (see Figure 3). From this information we will be able to calculate the foil thickness in the chamber and compare the theoretical and experimental scattering cross sections.

Equipment
1. Low Voltage Accelerator
2. Scattering Chamber
3. Solid State Charged Particle Detector
4. Detector Bias Supply
5. High Resolution Pre-amplifier
6. Spectroscopy Amplifier
7. Current Integrator
8. Multichannel Analyzer
9. Thin foils for thickness measurements

Procedure
1. Set up the electronics as shown in Figure 3. With an alpha source and a pulse generator, calibrate the solid state detector so that the output pulses range up to 200 keV. This procedure was outlined in an earlier experiment in this manual. We are assuming that the maximum energy of the accelerator which is available for this experiment is 200 keV.
Figure 3
Electronics for thin film measurement by elastic ion scattering.

Figure 4
Artist's concept of a scattering chamber for thin film scattering measurements.
2. Place the target to be studied in the target position. Let's assume that it is a thin gold foil (~100 μg/m²). Evacuate the scattering chamber and adjust the beam of the accelerator so that it will bombard a spot in the center of the target with an area of about 1 mm². Set the movable detector at an angle \( \theta = 40^\circ \) and observe the pulse height spectrum that is being accumulated in the multichannel analyzer. Figure 5 shows a typical spectrum that was obtained in a similar experiment. Note that the only group that is seen is a single elastically scattered peak.

![Pulse height spectrum for the \( ^{12}\text{C}(p,p)^{12}\text{C} \) reaction at \( E_p = 150 \text{ keV} \) and \( \theta_L = 30^\circ \).](image)

3. Change the accelerator energy from 200 keV to 150 keV and repeat the measurements in 2. Repeat at an incident beam energy of 100 keV.

4. Change to your second target and repeat Procedures 2 and 3.
Data Reduction

Exercise (a)

From the multichannel readout at 200 keV, sum under the scattered peak and subtract the estimated background. Enter this value as (E-β) in Table I. Record the channel number of the peak. Repeat for the other entries in Table I.

Table I

Foil Thickness Measurement for Protons on Gold, θ = 40°

<table>
<thead>
<tr>
<th>Incident Energy (keV)</th>
<th>E-β Peak</th>
<th>Peak Channel</th>
<th>Number of Incident Protons</th>
<th>ΣR Theory</th>
<th>Foil Thickness</th>
</tr>
</thead>
<tbody>
<tr>
<td>200</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>150</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>100</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

Figure 6

Theoretical and measured energies of 150 keV protons scattered from a thin carbon target.
Exercise (b)

From your energy calibration curve for the solid state detector, determine the energy of the peak for each of the runs. Since we are scattering from a heavy nucleus like gold, the kinematic energy loss is small and the scattered energy at 40° is approximately the energy of the accelerator. However, for lighter targets the situation is different. The incident projectile may experience rather large kinematical energy losses as well as energy losses just due to finite target thicknesses. Figure 6 shows a plot of what would be expected from 150 keV protons from a thin carbon target. Note that at a scattering angle of 40° the scattered proton has a theoretical scattered energy of 140 keV. The measured energy is about 115 keV. The dE of 25 keV is the average energy that the proton loses in going through the carbon target. Although there is little collision energy loss with a gold target, there is, of course, an energy loss due to the thickness of the gold foil as with carbon.

Exercise (c)

Calculate the target thickness for each of the runs as follows: the Rutherford scattering cross section, is given by the following theoretical formula:

\[
\frac{d\sigma}{d\Omega} \left( \text{millibarns per steradian} \right) = 1.296 \left( \frac{Z_1 Z_2}{E_{\text{lab}}} \right)^2 \left( \frac{1}{\sin^4 \theta_{\text{lab}}} \right) 
\]

where \( Z_1 = \) charge on the incident particle (one for protons)

\( Z_2 = \) charge on the target nucleus (79 for gold)

\( E_{\text{lab}} = \) incident energy in MeV

\( \theta_{\text{lab}} = \) measured angle (40° in our case)

The measured cross section is given by:

\[
\frac{d\sigma}{d\Omega} \left( \text{cm}^2 \text{ per steradian} \right) = \frac{(\Sigma - \delta)}{N n \Delta \Omega} 
\]

where \((\Sigma - \delta) =\) the sum under the scattered peak minus the background

\( N = \) number of target nuclei/cm²

\( n = \) number of incident protons

\( \Delta \Omega = \) solid angle in steradians of your detector

It can now be assumed that Eq (1) equals Eq (2), hence:

\[
\frac{d\sigma}{d\Omega_R} \times 10^{-27} = \frac{(\Sigma - \delta)}{N n \Delta \Omega} 
\]
Therefore

\[ N = \frac{(E - \beta)}{\frac{d\sigma}{d\Omega} n\Delta\Omega \times 10^{-27}} = \frac{\text{number of target nuclei}}{\text{cm}^2} \]  \tag{4}

and

\[ N = \frac{\text{gm/cm}^2 \text{ of target}}{\text{atomic weight}} (6.023 \times 10^{23}) \]

Therefore

\[ (\text{gm/cm}^2 \text{ of target}) = \frac{N \text{ (atomic weight)}}{6.023 \times 10^{23}} \]  \tag{5}

Fill in the foil thicknesses in Table I from Eq (5). Figure 7 shows that for gold our assumption that the measured cross section was equal to the theoretical value is a good one. In the figure \( \sigma_R \) is the theory and \( \sigma_{\text{exp}} \) is the experimental value. At 40\(^o\) the ratio of these two is one.

Figure 7

Ratio of the Rutherford experimental cross section to the theoretical value.

Exercise (d) Optional

If you wish, you can verify that the measured cross section agrees with the theory at other angles. We are assuming here that you have already determined the target thickness. Figures 8 and 9 show the agreement between the experiment and theory for the case of deuterons on gold and deuterons on boron. It should be obvious that this experiment can easily extend to a senior research project. In order to aid the student who wishes to follow either experimental or theoretical work on this subject we have included an abundant supply of references.
Figure 8
Measured and theoretical Rutherford scattering cross section for deuterons on gold.

Figure 9
Measured and theoretical Rutherford cross sections for deuterons on boron.
Post-Test

1.1 Why can only the number of particles passing through a thin foil and striking the Faraday cup be used to determine the number of incident particles? Why shouldn't one consider those incident particles that are scattered from the foil?

1.2 What is the thickness in cm of a 100 μgm/cm² thick gold foil? in inches? in microns (1 micron = 10⁻⁶ m)?

1.3 If the theoretical Rutherford differential scattering cross section for protons on gold is 63.4 b/sr, what would the value be for protons on silver? for protons on lead? If the proton energy was changed from 2 MeV to 1 MeV, what would the values of the cross sections be for the above targets?

1.4 Assuming a 3 microampere beam of 200 keV protons strikes a Faraday cup at the end of a beam line, how much power must be dissipated?

1.5 For a charged particle passing through a magnetic field, derive an expression for the radius of curvature as a function of magnetic field strength, B; the rest mass energy, mc²; the kinetic energy, T; and the charge of the particle, q.

Computer Programs

GAUSS-6 can be used to find the centroids of the pulser calibration spectra. It can also be used to find the elastic scattered peak position. LINEAR-6 can be used to make the calibration curve. The "Rutherford scattering" program will calculate the theoretical Rutherford scattering cross section for the experiment.

Additional References


Experiment 2

X-Ray Production Studies with a Small Accelerator

Objective

To study the production of characteristic X rays by-proton bombardment with a small Cockcroft-Walton or Van de Graaff accelerator.

References


Introduction

Many experiments that are done with accelerators are complicated either electronically or experimentally. This experiment doesn't suffer from either of these problems. Experimentally the problem is quite simple. A proton beam (see Figure 1) is allowed to impinge on the sample to be studied. The protons make a coulomb type interaction in the sample and, hence, remove a tightly bound electron (K or L) from one of the target atoms with the result that characteristic X rays are produced. The X ray can be measured either with a proportional counter, a Si(Li) or Ge(Li) detector as shown in Figure 1. Figure 2 shows a photograph of an Si(Li) X-ray detector and simple beam tube arrangement. In this figure the detector window is separated about 2 mm from the beam tube beryllium window. Shown also in the figure are the electronics for these measurements. There is a slight problem associated with bringing the X rays out of the accelerator beam tubing. This can be done by sealing a 1.25 x 10^-2 cm beryllium exit window onto the
Figure 1
Electronics for charged particle induced X-ray studies with a Ge(Li) detector.

beam tubing. Once the X-rays are produced, the rest of the counting procedures etc. are for the most part as we have already outlined in the previous X-ray fluorescence experiments.

The beam current for these experiments is usually kept to about 1 µAmp. The targets to be studied can either be infinitely thick to the beam of protons or thin targets.

There is no radiation hazard produced at the target when a low (less than 1 MeV) energy beam strikes a metal target. The best targets to study at low energies are the elements calcium through zinc for K lines and elements heavier than tin for L lines.

There are several points of interest in these experiments that should be mentioned. The first is that there is a considerable amount of interest in regard to the actual magnitude of the cross sections for these interactions. The simple plane wave Born approximation which is normally used to describe an interaction of this type has its shortcomings at these low energies. Secondly, there has recently been a great deal of interest expressed in using accelerators in this manner as an analytical probe to study surface phenomena. The best example is perhaps the semiconductor industry whose main interest is in ion implanted semiconductors. For example some of these devices are manufactured by implanting zinc ions into a silicon matrix. The questions to be answered are: how uniform is the implant and what is the depth of the implant. Both of these
Figure 2

Beam Tube, X-Ray Detector and Electronics for X-Ray Measurements
problems can be solved with charged particle induced X-ray measurements. The uniformity can be determined by studying the number of Zn X rays that are produced as a function of where the beam is striking the sample. The depth can be measured by observing the number of X rays produced as a function of bombarding energy. The lower energy ions only penetrate a few Angstroms into the surface and hence, a plot of the Zn Kα X-ray yield as a function of beam energy will give the depth of the implant.

Equipment

1. Low Energy Accelerator with X-Ray Target Assembly; Current Digitizer and Scaler
2. Si(Li) X-Ray Detector
3. Detector Bias Supply
4. Spectroscopy Amplifier
5. Multichannel Analyzer
6. Calibration Sources as follows: (all should be standard ±10%)
   a) 10 μCi $^{57}$Co
   b) 1 μCi $^{241}$Am
   c) 10 μCi $^{54}$Mn
   d) 10 μCi $^{51}$Cr

Procedure

1. Place the 10 μCi standard source in the position where the target will be when being bombarded with the beam. Accumulate a spectrum for a time period such that reasonable statistics are obtained in the calibration lines of interest. Repeat for the other standard sources listed in Table I. Fill in the peak channel information in the table.

2. Remove the radioactive sources and place a thin iron foil in the target positions. Set the beam current of the accelerator at around 500 nAmps and allow it to impinge on the target with an incident energy of 150 keV. Collect a spectrum for a long enough period of time to get reasonable statistics under the Fe Kα peak. Integrate the beam current for each run. Figure 3 shows a typical spectrum that was collected under similar conditions. Readout the multichannel analyzer. Repeat for the complete range of energies available with your accelerator (in 50 keV energy increments).
Table I

Energy & Efficiency Calibration of Accelerator Counting Geometry

<table>
<thead>
<tr>
<th>Radioisotope</th>
<th>Photon Energy (keV)</th>
<th>Peak Channel</th>
<th>Photons/sec Measured</th>
<th>Photons/sec Theory</th>
<th>Efficiency</th>
</tr>
</thead>
<tbody>
<tr>
<td>$^{57}$Co</td>
<td>6.40</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>$^{57}$Co</td>
<td>7.056</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>$^{54}$Mn</td>
<td>5.411</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>$^{51}$Cr</td>
<td>4.949</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>$^{241}$Am</td>
<td>13.9</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

Figure 3

Fe Kα spectrum produced by proton bombardment.
3. Repeat the measurements made in Procedure 2 for other thin targets. Figures 4 and 5 show a variety of other targets that were bombarded with a 350 keV proton beam from an educational Van de Graaff accelerator.

![Figure 4](image)

**Figure 4**

Characteristic X-ray spectra of Mn, FeSO₄, Ni, and Cu. Note: indicated energies are keV.
Figure 5

X-ray production spectra of KCl and Sc on copper backing.
Data Reduction

Exercise (a)

From the peak channel information recorded in Table I, plot a calibration curve. Find the slope of the calibration curve and determine the resolution of each of the peaks listed in the table.

Exercise (b)

Sum under the K group in the spectra collected in 1 and divide by the time and thus obtain the measured photons/sec in Table I. From the activities of the sources and the decay scheme information (see appendix), calculate the number of photons/sec that each source emits. Enter these values in Table I. Calculate the efficiency of the system which is the ratio of photons/sec measured to the theoretical count rate. Plot a curve of efficiency versus photon energy for your target configuration. Figure 6 shows a similar curve that was measured for an accelerator detector arrangement.

Figure 6

Efficiency curve for a Si(Li) detector with a 2.5 x 10^-2 cm Mylar entrance window.
Figure 7 shows an electronic block diagram that would be used for this experiment if a proportional counter were used instead of a Si(Li) X-ray detector.

![Diagram of electronic block diagram](image)

Figure 7

Charged particle induced X-ray studies with a proportional counter.

Exercise (c)

For each of the spectra collected, calculate the absolute cross section. This is done according to the following:

\[
\sigma_X \text{ (cm}^2\text{)} = \frac{(I-\beta)}{NN_0} \tag{1}
\]

where \((I-\beta)\) = sum under the \(K_{\alpha} + K_{\beta}\) groups in the spectra

\(N = \text{number of target nuclei/cm}^2 \)

\(N_0 = \text{number of incident protons} \)

\(\varepsilon = \text{efficiency obtained from Exercise (b)} \)

Calculate \(\sigma_X\) for all of the spectra collected. Plot \(\sigma_X\) versus energy for each of the elements studied.

Discussion

This kind of project makes an excellent senior research thesis. A search of the literature will quickly reveal that it is possible to do unique original work in this area. Projects of this type can be used by students as a basis for papers presented at Academy of Science meetings and school seminars.
Post-Test

2.1 Why do X-ray energies increase uniformly with atomic number \( Z \), while the energies of optical lines are so non-uniform even for adjacent elements in the periodic table?

Computer Programs

GAUSS-6 can be used to find the centroids of the peaks. LINEAR-6 can be used to least squares fit the calibration data.

Additional References

1. J. Chadwick, "The \( \alpha \) Rays Excited by the \( \beta \) Rays of Radium," Phil. Mag. 24, 594 (1912).


Pre-Test Answers

1.1 1 keV = 1000 eV
1 MeV = 10^6 eV
1 GeV = 10^9 eV

1.2 1 eV = 1.602 x 10^{-19} erg
1 eV = 1.602 x 10^{-19} joules

2.1 \( f = \frac{E}{\hbar} \)
\[ = \frac{11.92 \times 10^3 \text{ eV}}{6.625 \times 10^{-27} \text{ erg-sec}} \times \frac{1.602 \times 10^{-12} \text{ erg}}{\text{eV}} \]
\[ = 2.88 \times 10^{18} \text{ sec}^{-1} \]
\[ \lambda = \frac{c}{f} \]
\[ = \frac{3 \times 10^{10} \text{ cm/sec}}{2.88 \times 10^{18} \text{ sec}^{-1}} \]
\[ = 1.04 \times 10^{-8} \text{ cm} \]

3.1 The force is given by the familiar coulomb's law for point charges,
\[ F = \frac{1}{4\pi\varepsilon_0} \frac{q_1 q_2}{r^2} \]
where \( q_1 \) and \( q_2 \) are the electronic charges, \( r \) is the distance separating the proton and the electron and \( \varepsilon_0 \) is the permittivity of free space and has the value \( \varepsilon_0 = 8.854 \times 10^{-12} \) coul^2/n-m^2.

The distance \( r \) is radius of the smallest Bohr orbit and is given by
\[ r = \frac{\hbar^2}{m e^2} \]

Multiplying both numerator and denominator by \( c^2 \) and rearranging gives
\[ r = \frac{\hbar^2}{me^2} \cdot \frac{c^2}{\epsilon^2} \]
\[ = \frac{\hbar c}{mc^2 \left( \frac{e^2}{\hbar c} \right)} \]
\[ r = \frac{197.32 \text{ MeV} - \text{fermis}}{0.511 \text{ MeV} \cdot \frac{1}{137}} \]
\[ r = 0.529 \times 10^{-8} \text{ cm.} \]

Hence,
\[ F = \frac{1}{4\pi (8.854 \times 10^{-12} \text{ coul}^2/\text{n-n}^2)} \cdot \frac{(1.6 \times 10^{-19} \text{ coul})^2}{(0.529 \times 10^{-10} \text{ m})^2} \]
\[ = 8.222 \times 10^{-8} \text{ n} \]

4.1 The magnetic force is given by the expression,
\[ F = qvB. \]

The velocity of a 150 keV proton is
\[ v = \left( \frac{2E}{m} \right)^{\frac{1}{2}} \]
\[ = \left[ \frac{2(150 \times 10^3 \text{ ev})(1.6 \times 10^{-19} \text{ joule/ev})}{1.67 \times 10^{-27} \text{ kgm}} \right]^{\frac{1}{2}} \]
\[ = 5.36 \times 10^6 \frac{\text{ m}}{\text{ sec}}. \]

Therefore, the magnetic force is
\[ F = (1.6 \times 10^{-19} \text{ coul})(5.36 \times 10^6 \text{ m/s})(0.6 \times 10^4 \text{ gauss}) \]
\[ \times (10^{-4} \frac{\text{ weber}}{\text{ m}^2 \text{ gauss}}) \]
\[ = 5.15 \times 10^{-13} \text{ newton} \]

5.1 \( \frac{d\psi}{d\Omega} \) Rutherford = 1.296 \( \left( \frac{Z_1 Z_2}{E} \right)^2 \frac{1}{\sin^n \left( \frac{\theta}{2} \right)} \)
Post-Test Answers

1.1 Only a very small fraction of the incident particles are scattered away from the forward direction through interactions with nuclei in a thin foil. For this reason, cross sections or the probabilities for scattering are measured in units of barns \(10^{-24} \text{ cm}^2\).

1.2 \(\rho t = 100 \ \mu \text{gm/cm}^2\)

\[
t = \frac{100 \times 10^{-6} \ \text{gm/cm}^2}{19.32 \ \text{gm/cm}^3}
\]

\(= 5.18 \times 10^{-6} \ \text{cm}\)

\(= 2.04 \times 10^{-6} \ \text{inches}\)

\(= 0.0518 \ \text{microns}\)

1.3 \(\frac{d\sigma}{d\Omega} \propto \left(\frac{Z_1 Z_2}{E}\right)^2\)

\[
\frac{d\sigma}{d\Omega} \text{ (gold)} = 63.4 \ \text{b/sr}
\]

\[
\frac{d\sigma}{d\Omega} \text{ (silver)} = \left(\frac{Z_2 \text{(silver)}}{Z_2 \text{(gold)}}\right)^2 \times 63.4 \ \text{b/sr}
\]

\(= \left(\frac{47}{79}\right)^2 \times 63.4 \ \text{b/sr}\)

\(= 22.4 \ \text{b/sr}\)

\[
\frac{d\sigma}{d\Omega} \text{ (lead)} = \left(\frac{82}{79}\right)^2 \times 63.4 \ \text{b/sr}
\]

\(= 68.3 \ \text{b/sr}\)
For 1 MeV incident protons

\[ \frac{d\sigma}{d\Omega} \text{ (gold)} = \left( \frac{2}{1} \right)^2 63.4 \text{ b/sr} \]

\[ = 4 \times 63.4 \text{ b/sr} \]

\[ = 253.6 \text{ b/sr} \]

\[ \frac{d\sigma}{d\Omega} \text{ (silver)} = 89.6 \text{ b/sr} \]

\[ \frac{d\sigma}{d\Omega} \text{ (lead)} = 273.2 \text{ b/sr} \]

1.4 Power = \( (3 \times 10^{-6} \text{ ampere})(0.2 \times 10^6 \text{ eV}) \)

\[ = 0.6 \text{ watts} \]

1.5 From Newton's second law

\[ ma = F \]

\[ \frac{mu^2}{r} = quB \]

\[ r = \frac{mu^2}{quB} \]

\[ = \frac{p}{qB} \]

The total relativistic energy of the charged particle may be written

\[ m^2c^4 = p^2c^2 + m_0^2c^4 \]

or

\[ p = \frac{1}{c} \left[ m^2c^4 - m_0^2c^4 \right]^{1/2} \]

But the total energy may be written in terms of the kinetic energy and the rest mass energy

\[ mc^2 = T + m_0c^2 \]

Squaring both sides of this expression gives

\[ m^2c^6 = T^2 + 2m_0c^2T + m_0^2c^4 \]
Substituting in the expression for the momentum $p$, results in

$$p = \frac{1}{\gamma C} \left[ T^2 + 2m_o C^2 \right]^{\frac{1}{2}}$$

Hence, the radius of curvature may be written

$$r = \frac{1}{\gamma C B} \left[ T^2 + 2m_o C^2 \right]^{\frac{1}{2}}$$

2.1 The energy of the characteristic X rays depends on the binding energies of the electrons in the inner shells. With increasing atomic number $Z$, these binding energies increase rather uniformly with increased nuclear charge and are very little affected by the periodic changes in the number of electrons in the outer shells.
APPENDIX LISTINGS

I Regulations Concerning Safe Radioisotope Use and a Nonexhaustive Radioisotope Vendor File

II Programs Developed for Reducing Data in this Manual

III Reactor and Accelerator Facility Sharing Program

IV Absolute Disintegration Rates for Some of the Most Commonly Used X-Ray and Gamma Ray Calibration Sources

V X-Ray Critical-Absorption and Emission Energies in KeV

VI Relative Sensitivities of Elements to Neutron Activation (Thermal)

VII Major Gamma Rays Observed in Neutron Activation Analysis

VIII General Textbook References for Radioisotope Experiments

IX Government Programs that are Designed to Help Colleges and Universities with the Funding of Educational Scientific Equipment
APPENDIX I

Regulations Concerning Safe Radioisotope Use and a Nonexhaustive Radioisotope Vendor File

All users of radioisotopes must follow regulations concerning safe radioisotope use. A copy of Code of Federal Regulations, Title 10 - Atomic Energy, Chapter 1, Part 20 - Standards for Protection Against Radiation, and Part 30 - Licensing of Byproduct Materials should be obtained by all potential users. A free copy can be obtained by writing:

Administrator for Nuclear Energy
Energy Research and Development Administration
Washington, D. C. 20545

An exhaustive list of manufacturers that supply radioisotopes for educational use can be found in the Isotope Index, J. L. Sommerville, Ed., Scientific Equipment Company Publications Department, P. O. Box 19086, Indianapolis, Indiana 48219 (Price $2.00). Listed below are some names and addresses of a few companies from the Index. All of these companies will be glad to furnish upon request descriptive literature with regard to their isotopes.
Appendix II

Programs Developed for Reducing Data in this Manual

General Remarks

These programs have been written for the reduction of experiments in this manual. In each case, adequate testing has been done to assure the author that the program works... at least when he runs it! The programs have been written for the most part in Basic Language. It should be quite easy to modify a given program so that it can be used on the computer in your school.

If programs can be used to reduce the data in these experiments, they should be, since this makes the whole experience more meaningful.

There are descriptions, instructions, and examples on the pages that follow. Examine them carefully.

There are many ways for you to louse-up a program...!

The most obvious of these is to input bum data. No matter how clever a programmer becomes, he can never provide for erroneous input... and the poor computer is too dumb to know the difference. It treats everything as Gospel. The result will be garbage... and you'd better be able to recognize it before you go bragging about your most recent discovery.

The best place for bum data to hide is in BIG data blocks. Edit these carefully with particular attention to the placement of commas. Omission of one comma can be a disaster...! Be sure that a decimal has not been substituted for a comma or vice versa. Transposed numbers represent still another trap and are among the more difficult input errors to find.

It is imperative that the data be placed in the order described in the
Edit your input to running programs with equal care. Mostly, an error is detected before the RETURN key is pressed while the RUBOUT key can come to your rescue. Once RETURN is pressed, there is no recovery. Abort the run with CTRL/C and try again.

A truly effective way for user to blow a program is to DELETE a line number after loading the program. This can be done quite innocently when he starts typing data before listing a line number and DATA statement... now suddenly realizing his mistake, he presses RETURN to try again. ZAP...! There goes a line whose number happens to coincide with the data. Sometimes this will be detected by the compiler... most often not.

When you decide you've loused up a data line so badly that it must be retyped... use the ALT-MODE key instead of the RETURN. Don't try to patch a mutilated program. It is better to reload it and start again.

Lots of Luck...!!!
PROGRAM NAME: LINEAR 6

This program computes a linear least-squares fit to a set of variables of X and Y.

The slope, intercept and their standard deviations are provided. The data is then examined to locate points which are statistically invalid by application of Chauvenet's Criteria.

The user has the option of inputting values of the independent variable to obtain the corresponding dependent variable at the conclusion of the run.

It is necessary to prepare a data block before running the program.

INPUT

Instructions for preparing the data block . . ., etc., (see SPEC)

Input the number of data pairs when asked for by the running program.

OUTPUT

1. The slope and intercept
2. The standard deviation of each
3. Chauvenet's Reject Level in standard deviation
4. The Residuals
5. A refitting if point is rejected

OPTIONS

1. Input a value of X to obtain the corresponding Y
PROGRAM NAME: LINEAR 6

RATIONALE

Slope, \( M = \frac{\Sigma xy - \Sigma x \Sigma y}{\Sigma x^2 - (\Sigma x)^2/N} \)

Intercept, \( B = (\Sigma y - m \Sigma x)/N \)

Determinant, \( D = \Sigma x^2 - (\Sigma x)^2/N \)

Intercept Error, \( Y_3 = [(\Sigma y^2 - (\Sigma y)^2/N - DM^2)/(N - 1)]^{1/2} \)

Slope Error, \( M_2 = Y_3/\sqrt{D} \)

Chauvenet's Reject Level, algorithm
\[ C = 2 + \ln(N/11), \text{ sigma} \]

Residual
\[ D = (Y - B - MX)/Y_3, \text{ sigma} \]
NEW CR CLD--C
CLD PROGRAM NAME--LINEAR 6

READY

LIST 1, 250
1 REM INSTRUCTION FOR LINEAR LEAST-SQUARES PROGRAM
2 REM
3 REM INSERT DATA PAIRS IN LINES 501-600, SEQUENTIALLY
4 REM EXAMPLE:
5 REM 501 DATA X1, Y1, X2, Y2
6 REM 502 DATA X3, Y3, X4, Y4, X5, Y5
7 REM IT IS IMPORTANT TO SEPARATE EACH ENTRY WITH A COMMA
8 REM IT IS ALSO IMPORTANT TO USE THE COMMA AT THE END OF LINE
9 REM BE SURE TO COUNT YOUR ENTRY PAIRS.
10 REM. THERE MUST BE MORE THAN TWO!!
11 PRINT "LINEAR LEAST-SQUARES PROGRAM"
12 PRINT
13 PRINT "THE NUMBER OF DATA PAIRS IS ":
14 INPUT N
15 IF N<2 THEN 50
16 PRINT
17 PRINT "MORE DATA, PLEASE!!"
18 PRINT "TWO POINTS DETERMINE A STRAIGHT LINE!"
19 STOP
20 FOR I=1 TO N
21 READ X, Y
22 LET Y1=X+Y
23 LET Y2=Y+Y
24 LET S=S+XY
25 LET Y1=Y1+Y
26 LET Y2=Y2+Y
27 NEXT I
28 COSP=140
29 GOTO 30
30 LET I=S-X1*Y1/N
31 LET D=Y2-X1*Y1/N
32 LET X=S/N
33 LET P=(Y1-X*Y1)/N
34 PRINT "SLOPE","INTERCEPT"
35 LET Y3=SOL(Y3+D*M)/N)
36 PRINT "DEV", "DEVIATION ", Y3
37 PRINT
38 RETURN
39 END
LIST 250, 610

250 STOP
260 LET C=2+LGL(N/11)/2.3
270 PRINT "REJECT LEVEL: "#SIGMA""
275 PRINT
280 RESTORE
285 PRINT "RESIDUALS:"
290 PRINT
295 FOR I=1 TO N
300 READ X, Y
305 LET D=(Y-B-A*X)/Y3
310 PRINT D1
315 IF ABS(D)<C THEN 360
320 PRINT ",";"POINT ("X;Y") IS A STINKER....WILL DISCARD"
325 LET H=H+1
330 LET X1=X1-X
335 LET Y2=Y2-Y
340 LET S=S-X*Y
345 LET Y1=Y1-Y
350 LET Y2=Y2-Y
355 GOTO 365
360 PRINT
365 NEXT I
370 IF H=3 THEN 387
375 LET N=N-R
380 PRINT "WITH "#N"DATA PAIRS....."
385 GOSUB 140
390 PRINT
395 PRINT "YOU MAY NOW INPUT X VALUES"
399 PRINT
400 PRINT "WHEN X IS 0"
405 INPUT X
410 PRINT ", Y IS "+B"
415 GOTO 395
420 DATA 31.508, 511, 40.986, 661.6, 55, 985, 898.01
425 DATA 58, 965, 84, 02, 1274.52, 116, 019, 1836.08
430 DATA 250
440 END

READY
PROGRAM NAME: GAUSS 6

This program computes the centroid of a Gaussian distribution and continues with a least-squares fit of the data.

While primarily intended for use in analyzing the photopeaks in multichannel scintillation spectra, the program also serves Si(Li) and Ge(Li) spectra, provided there are a sufficient number of channels in the peaks.

Selected data from the peak is read from a data block and there are options available during a run.

INPUT

Directions for preparing the data are contained in the form of remarks in the program and can be called by typing: LIST 1,8

OPTIONS

1. Stripping correction:
   Input the count in the channels just above the peak that represent the average of the continuum upon which the peak is resting. This will be subtracted from the count in each channel used in fitting the peak. Input zero if desired.

2. Counting Interval:
   When asked, input the live time used in making the spectrum. The count-rate of the peak will then be computed.

3. Exit with CTRL/C

OUTPUT

1. The peak centroid
2. The peak maximum
3. The peak integral
4. The resolution
5. The peak counting rate
RATIONALE

\[ x_0 = \frac{\Sigma xy^2}{\Sigma y^2} \]
\[ y = y_0 \exp\left[ b (x - x_0)^2 \right] \]
\[ b = \frac{\Sigma xy - \Sigma x \Sigma y / N}{\Sigma x^2 - (\Sigma x)^2 / N} \]
\[ y_0 = \exp\left[ \frac{\Sigma y - \Sigma E X / N}{N} \right] \]
\[ \Sigma y = y_0 \sqrt{\pi / b} \]
\[ \rho = 2 \cdot \frac{x_0}{x} \sqrt{-\ln 0.5} \]

- \( x \): channel number
- \( y \): a count
- \( x_0 \): centroid of peak
- \( X \): \((x - x_0)^2\)
- \( Y \): \(\ln y\)
- \( N \): the number of data pairs
- \( b \): the Gaussian constant
- \( y_0 \): the count at the centroid, \( x \)
- \( \Sigma y \): the integral of the Gaussian
- \( \rho \): the resolution
LIST
1 REM GAUSSIAN LEAST-SQUARES PROGRAM
2 REM
3 REM PREPARE DATA BLOCK BEGINNING WITH LINE 400
4 REM XI IS CHNL NO. AT HALF-HT, LOW SIDE OF PEAK
5 REM YI IS COUNT IN CHNL XI
6 REM 400 DATA XI, Y1, Y2, Y3, ..., YN, 0
7 REM YN SHOULD BE ABOUT THE SAME AS Y1
8 REM TERMINATE DATA STRING WITH A ZERO'
10 PRINT "STRIPPING CORRECTION";
15 INPUT C
20 READ X
25 READ Y
30 IF Y=0 THEN 55
32 LET Y=Y-C
35 LET Y2=Y2+Y1*2
40 LET Y3=Y3+X*Y1*2
45 LET X=X+1
50 GOTO 25
55 LET X3=Y3/Y2
58 PRINT
60 PRINT "CENTROID IS" INT(100*X3+5)/100
70 RESTORE
75 READ A
80 READ Y
85 IF Y=0 THEN 150
90 LET Y=LOG(Y-C)
100 LET X=(A-X3)*2
110 LET X1=X1+X
115 LET X2=X2+X*2
120 LET S=S+X*Y
125 LET Y1=Y1+Y
130 LET A=A+1
135 LET N=N+1
140 GOTO 80
150 LET M=S-X1*Y1/N
155 LET D=X2-X1*2/N
160 LET H=M/D
165 LET Y0=(Y1-B*X1)/N
180 LET r=3.14159
190 LET H=INT(EXP(Y0))
200 PRINT "MAX. PEAK HT."
205 LET S=H*SQR(ABS(P/B))
210 PRINT "INTEGRAL OF PEAK" INT(SRQ(LOG(5)/B2)*2/X3)
220 PRINT "RESOLUTION" INT(100*R+S)/100"
300 PRINT
310 PRINT "COUNTING INTERVAL (MIN)"
320 INPUT T
PROGRAM NAME: FLUX

This program computes the neutron flux of an irradiation facility on the basis of the activity induced in a monitor.

All data is input into the running program and there is an option concerning the measurement of the absolute activity of the monitor. It is, however, assumed that 3x3-inch NaI(Tl) scintillation detectors will be used.

INPUT

1. Element used for flux monitor
2. Isotope, half-life, units
3. Monitor weight, % element
4. Term of irradiation
5. Photopeak count observed
6. Live time
7. Wait time
8. Clock time
9. Energy of gamma emission measured
10. Absolute intensity of emission
11. Reaction cross section
12. Isotopic abundance of target nuclide
13. Atomic weight of element

OUTPUT

...the computed neutron flux, n/sqcm-sec

OPTION

At the beginning of a run, answer "Y" or "N" regarding the use of a Cs-137 standard.

A "Y" response permits input regarding the Cs-137 standard

1. The net CPM in the photopeak
2. The number of 662 KeV photons/min

The counting yield is computed and the program proceeds to the input listed above.

A "N" response assumes that the low-level counting station is being used for measurement and the calibration for this facility is applied.
LIST
20 PRINT "LCS CS-137 STANDARD COUNTED(\% OR N)";
25 INPUT A$,
30 IF A$="y" THEN 400
35 LET K=.089
40 PRINT
45 PRINT "ELEMENT USED FOR FLUX MONITOR";
50 INPUT E$
55 PRINT "ISOTOPE, HALF-LIFE, UNITS(MIN,HR)";
60 INPUT I$,T,U$
65 LET L=LOG(2)/L
70 PRINT
75 PRINT "MONITOR WI(MG),Z";
80 INPUT W,P0
85 PRINT "TERM OF IRRADIATION IN "US;
90 INPUT T1
95 LET S=1-EXP(-L*T1)
100 PRINT "CUMULATIVE OBSERVED";
105 INPUT N
110 PRINT "LIVE TIME(MIN)";
115 INPUT T2
120 PRINT "TIME IN "US;
125 INPUT T3
130 IF US="MIN" THEN 170
140 LET R=N/(T3*EXP(-L*T1))
150 GOTO 200
170 PRINT "CLOCK TIME(MIN)";
175 INPUT T2
180 LET N=N*T2/13
185 LET N=EXP(-L*13-12)
200 PRINT
230 PRINT "GAMMA ENERGY MEASURED(KEV), ABS.INT.(%)";
235 INPUT E$
240 LET E=E/1000
250 LET E=E/.05
255 LET A=R/(Y*1)*100/60.
260 PRINT "CROSS SECTION(BARNS)";
265 INPUT C
270 PRINT "ABUNDANCE OF TARGET NUCLIDE(%)";
275 INPUT C
280 PRINT "ATOMIC WT. OF "E$
285 INPUT W
290 LET N=W*C/100*P0/100*1.3E+20
295 LET F=A/N*C*1E-24.5
300 PRINT
310 PRINT "...FLUX IS"F" NEUTR./SQCM-SEC";
320 PRINT
340 PRINT "GAM IN SOICPEAK";
345 INPUT G
350 PRINT "662 KEV PHOTONS/\%";
345 INPUT G
RUTHERFORD SCATTERING* PROGRAM

10 PRINT "RUTHERFORD SCATTERING PROGRAM"
15 PRINT
20 PRINT "Z1,Z2";
30 INPUT Z1,Z2
40 PRINT "ENERGY";
50 INPUT E
60 PRINT "ANGLE INCREMENT";
70 INPUT D
75 PRINT
80 PRINT "ANGLE","DS/DO"
100 FOR A=5 TO 18 STEP D
110 PRINT A,
115 LET X=A/114.6
130 PRINT 1.296*(Z1*Z2/E)^2/(SIN(X)^4)
140 NEXT A
150 END'

READY
Appendix III

Reactor and Accelerator Facility Sharing Program

The following is a list of colleges, universities, and institutes that have expressed a willingness to do collaborative work with their reactors and accelerators. If you are interested in going to these facilities for an experiment, tour, or demonstration, a letter of inquiry from your professor should be directed to the proper individual listed on the next few pages.

The authors wish to express their appreciation to ORTEC, Inc., Oak Ridge, Tennessee and to the Special Projects Office, Oak Ridge Associated Universities, for helping with the compilation of this information.
<table>
<thead>
<tr>
<th>Facility</th>
<th>Contact</th>
<th>Address</th>
<th>Facility Description</th>
</tr>
</thead>
<tbody>
<tr>
<td>University of Arizona</td>
<td>Dr. M. V. Lewis</td>
<td>Dept. of Nuclear Engineering, University of Arizona, Tucson, Arizona 85721</td>
<td>Atomic International, 10 MW, homogeneous reactor, 100 MW neutrons, Argonneau</td>
</tr>
<tr>
<td>Brigham Young University</td>
<td>Dr. Dwight R. Dixon</td>
<td>Brigham Young University, Provo, Utah 84601</td>
<td>AGW 21</td>
</tr>
<tr>
<td>University of California (Los Angeles)</td>
<td>Dr. Thomas E. Hulten</td>
<td>University of California, Los Angeles, California 90024</td>
<td>AGW 700</td>
</tr>
<tr>
<td>Catholic University</td>
<td>Dr. R. W. Deutsch</td>
<td>Catholic University, Washington, D.C. 20017</td>
<td></td>
</tr>
<tr>
<td>Colorado State University</td>
<td>Dr. H. G. Olson</td>
<td>Colorado State University, Boulder, Colorado 80309</td>
<td></td>
</tr>
<tr>
<td>Columbia University</td>
<td>Professor Edward Medlin</td>
<td>Columbia University, G.West 2000 Street, New York City 10027</td>
<td></td>
</tr>
<tr>
<td>Cornell University</td>
<td>Professor David D. Clark</td>
<td>Cornell University, Ithaca, New York 14853</td>
<td></td>
</tr>
<tr>
<td>University of Delaware</td>
<td>Dr. Bruce J. Leo</td>
<td>University of Delaware, Nuclear Engineering Department, University of Delaware, Newark, Delaware 07102</td>
<td></td>
</tr>
<tr>
<td>University of Florida</td>
<td>Dr. Richard W. England</td>
<td>University of Florida, Gainesville, Florida 32601</td>
<td>100 MW once-through reactor, heavy water moderated</td>
</tr>
<tr>
<td>Georgia Institute of Technology</td>
<td>Dr. Milton E. McClain</td>
<td>Georgia Institute of Technology, Atlanta, Georgia 30332</td>
<td></td>
</tr>
<tr>
<td>University of Illinois</td>
<td>Dr. Ronald P. Begley</td>
<td>University of Illinois, Nuclear Reactor Laboratory, Urbana, Illinois 61801</td>
<td>LEU, 200 MW, 1000 MW powerplant, 1000 MW reactor, heavy water, 1000 MW reactor, heavy water moderated</td>
</tr>
<tr>
<td>Iowa State University</td>
<td>Dr. Glenn Murphy</td>
<td>Iowa State University, Nuclear Engineering Dept., Ames, Iowa 50011</td>
<td></td>
</tr>
<tr>
<td>Kansas State University</td>
<td>Dr. Faron G. Resch</td>
<td>Kansas State University, Manhattan, Kansas 66507</td>
<td></td>
</tr>
<tr>
<td>University of Kansas</td>
<td>Professor R. B. Smeda</td>
<td>University of Kansas, Reactor Reactor Center, University of Kansas, Lawrence, Kansas 66044</td>
<td>Pool Type 10 MW</td>
</tr>
<tr>
<td>Lowell Technological Institute</td>
<td>Dr. Loretta F. Berman</td>
<td>Lowell Technological Institute, Lowell, Kansas 66507</td>
<td>LEU, 200 MW, 1000 MW, 1000 MW reactor, heavy water moderated, 1000 MW reactor, heavy water moderated</td>
</tr>
<tr>
<td>Marquette University</td>
<td>Dr. B. J. Richardson</td>
<td>Marquette University, 501 W. Wisconsin Ave, Milwaukee, Wisconsin</td>
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### REACTOR FACILITY SHARING PROGRAM (Continued)

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<td>Stanford University</td>
<td>Dr. Thomas J. Conklin</td>
<td>Nuclear Engineering Division, Stanford University, Stanford, California 94305</td>
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<td>Texas A&amp;M University Nuclear Science Center, University of Texas (Austin)</td>
<td>Dr. John D. Randall, Dr. Stephen J. Gage</td>
<td>College Station, Texas 77843</td>
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<td>AGN 201M (5 W)</td>
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<td>Dr. Andrew Robinson, Dr. J. Niven</td>
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### ACCELERATOR SHARING PROGRAM

The following is a list of colleges, universities, and institutes that have expressed a willingness to do collaborative work with their accelerators. Contact any of these facilities for details.

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<th>Facility Description</th>
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<td>Mississippi State University, State College, Albuquerque, NM 87106</td>
<td>PAM – Lateral Accelerator</td>
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<tr>
<td>Rensselaer Polytechnic Institute</td>
<td>Dr. J. L. Goodwin</td>
<td>Washington State University, Pullman, Washington 99163</td>
<td>PAM – Lateral Accelerator</td>
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<td>University of Utah</td>
<td>Dr. G. M. Wehry</td>
<td>University of Utah, Salt Lake City, Utah 84112</td>
<td>TRAC – Accelerator – 214</td>
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Note: The text contains some typographical errors and abbreviations, which may affect the accuracy of the table entries.
Absolute Disintegration Rates for Some of the Most Commonly Used X-Ray and Gamma Ray Calibration Sources

These two tables give the latest photon energy and photons emitted per decay for some of the most commonly used X-ray and gamma ray sources. The Reference list at the end of the Appendix gives the sources of data.

The author wishes to express his appreciation to Dr. Richard W. Fink, School of Chemistry, Georgia Institute of Technology, Atlanta, Georgia, for allowing us to use his data prior to publication.
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<tr>
<th>Nuclide</th>
<th>Daughter</th>
<th>Radiation</th>
<th>Photon Energy (keV)</th>
<th>Photon Emitted Per Decay</th>
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<tbody>
<tr>
<td>Am-241</td>
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<td>M X rays</td>
<td>3.30 (a)</td>
<td>0.0635 ± 0.0060 (b)</td>
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<td>Lα X rays</td>
<td>13.9</td>
<td>0.135 ± 0.003 (c)</td>
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<td>Lβ X rays</td>
<td>17.8</td>
<td>0.202 ± 0.004 (d)</td>
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<td>0.050 ± 0.001</td>
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<td>γ ray</td>
<td>26.4</td>
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<td>0.359 ± 0.006</td>
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<td>Fe-57</td>
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<td>γ ray</td>
<td>14.36</td>
<td>0.095 ± 0.002 (f)</td>
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<td>γ ray</td>
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<td>Co-60</td>
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<td>1332.49</td>
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<td>Cs-137</td>
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<td>Hg-203</td>
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### Table I, continued

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<td>Na-22</td>
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<td>1274.55</td>
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<td>T-1/2 = 950.3d</td>
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<td>Y-88</td>
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<td>T-1/2 = 1.066·10^3d</td>
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### Table II

**Standardization Sources**

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a. M x-rays from the decay of Am-241 are spread from about 3 to 5.6 KeV. Special caution should be exercised with germanium detectors since the K\textsubscript{a} and K\textsubscript{B} escape in germanium from the 13.9 KeV L\textsubscript{a} of Am-241 falls within the M x-ray region of Am-241.

b. Ref. 1
c. All values other than those footnoted are as given by IAEA, Vienna.
d. Ref. 2
e. Calculating using \( \chi/\gamma(14) = 5.82 \) from Ref. 3
f. Using the data listed in Ref. 4
g. Using \( \alpha_K \) and \( \gamma/\text{dis.} \) from Ref. 5 and \( \omega_K \) from Ref. 6.
h. Using \( K_B/K_a = 0.2370 \pm 0.0048 \) from Ref. 7.
i. The original values as supplied by IAEA, Vienna, have been corrected for the \( K_B/K_a \) intensity ratio given in Ref. 7.
j. Computed using an average value of \( P_K w_K = 0.250 \pm 0.002 \).
k. Computed using \( P_K w_K = 0.6340 \pm 0.0032 \) from Ref. 8.

a. All values given in this table are normalized to the 100.1 KeV \( \gamma \) ray.
b. An average of values given in Refs. 9 and 10.
c. An average of values given in Refs. 2, 9, and 10.
REFERENCES
(Appendix IV)


X-Ray Critical-Absorption and Emission Energies in keV

By S. FINE and C. F. HENDEE
Physics Laboratories
Princeton University
Princeton, New Jersey

Increased use of energy-proportional detectors for X-ray's has created a need for a table of energy values of K and L absorption and emission lines.

The table presented here includes all elements. Most values were obtained by a conversion to keV of tabulated experimental wavelength values (17) which are from previous energy-value compilations (2, 3). When new data were available these values were replaced by later work. Certain values were determined by interpolation, using Moxley's law. (All this is annotated in footnotes.)

The conversion equations relating energy and wavelength used are (6):

\[ E(\text{keV}) = \left( \frac{12395.1}{0.800017} \right) \lambda(\text{A}) \]

\[ E(\text{keV}) = \left( \frac{12394.1}{0.802026} \right) \lambda(\text{A}) \]

In computing values the number of places retained sufficed to maintain the uncertainty in the original source value. The values in the table have been listed uniformly to 1 keV. However, chemical form may shift absorption edges as much as 10-20 keV (4, 5).

To discover computational errors a fit was made to Moxley's law. In general the values were consistent, however there were a few irregularities due to the deviation of some input values (1). These were retained in the body of the table but a set of values calculated to fit better are footnoted.

BIBLIOGRAPHY

2. G. C. Pease, Phys. Rev. 73, 117 (1948).

The authors wish to express their appreciation to W. P. Smith for helpful suggestions and to H. Kupper for performing the computations in connection with this work.

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**For Z > 99, values without symbols are derived from (1). Values prefixed with a * sign are derived.**

1. Values from standard tables are used in the case of Z = 1. The values for Z = 2 are derived from the preceding standard values and elements beyond Z = 64 are calculated.


3. Calculated by interpolation where necessary.
APPENDIX VI

Relative Sensitivities of Elements To Neutron Activation (Thermal)

This tabulation should be quite useful to schools that have either an Isotopic Neutron Source (i.e. Pu-Be) or a small Cf-252 Neutron Source. The numbers are equally valid for thermalized accelerator neutron sources. Information in regard to unknowns which are given in the activation analysis experiments in this manual can also be extracted from this table.

Experimentally determined sensitivities are relative to Aluminum. Thus, if 1-milligram of Aluminum can be measured, then the irradiation and counting system is capable of measuring the listed quantities of elements in milligrams.

In each case, the reaction product and gamma energy have been selected which give the best interference-free sensitivity.

Irradiation to saturation is assumed for nuclides having half-lives in seconds or minutes. An overnight (16-hour) irradiation is assumed for the longer lived nuclides.

Measurements of the activities were made with NaI(Tl) scintillation detectors. Sensitivity was assigned on the basis of the amount of element required to provide a discharge count rate of 100 net counts per minute in the photopeak indicated for the product nuclide.

The listed relative sensitivities would be approximately the same with measurement using Ge(Li) detectors. Longer counting periods are made necessary because of lower efficiency; and, hence, very short-lived activities can be expected to show a decreased sensitivity. The higher resolution of the Ge(Li), however, is an advantage when interferences are present.
<table>
<thead>
<tr>
<th>At. No.</th>
<th>Symbol</th>
<th>Nuclide</th>
<th>Half-Life</th>
<th>Measured By</th>
<th>Relative Sensitivity</th>
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<td>-----------</td>
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<td>78</td>
<td>1.2</td>
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</tbody>
</table>
## NAA SENSITIVITY

### Elements and Sensitivity Levels

- **Low Sensitivity**: > 10
- **Medium Sensitivity**: 0.1 - 10
- **High Sensitivity**: < 0.1

### Diagram

A periodic table of elements is shown, with a grid labeled for sensitivity levels. The elements are color-coded to indicate their sensitivity level. The elements are arranged in the periodic table format, with elements such as H, He, Li, Be, etc., placed accordingly.
APPENDIX VII

Major Gamma Rays Observed in Neutron Activation Analysis

The following table includes only those nuclides most frequently observed as products of neutron bombardments. It is ordered according to increasing energy of a prominent gamma ray, designated as $E_1$, emitted in the decay of the nuclide.

If the nuclide is present, it is highly likely that $E_1$ will show an appreciable intensity in the spectrum and it thus serves as a key to the identity of the nuclide. The half-life and associated gammas should then be consulted for confirmation.

The last column shows the sensitivity relative to that for aluminum, assuming that an adequate irradiation has been performed. This too, can be of help in eliminating unlikely prospects and confirming an identification.

<table>
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<tr>
<th>Nuclide</th>
<th>$E_1$ (MeV)</th>
<th>Sensitivity Relative to Aluminum</th>
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...
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<th>T-1/2</th>
<th>E₂</th>
<th>E₃</th>
<th>E₄</th>
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<td>Hg-197</td>
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<td>1.2</td>
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<td></td>
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<td>1147</td>
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<td>308</td>
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APPENDIX IX

Government Programs that are Designed to Help Colleges and Universities with the Funding of Educational Scientific Equipment

Three agencies of the United States Government have had programs to assist colleges in the procurement of scientific equipment for instructional purposes. These programs are in addition to the various research grant programs conducted by a number of agencies that include the purchase of research equipment. Of course, use of such instructional equipment for student and staff research is encouraged, and it is important that the quality of the equipment be such that both functions may be served.

Atomic Energy Commission (Surplus Equipment Program)
For current information write to:
Administrator for Nuclear Energy
Energy Research and Development Administration
Washington, D. C. 20545

National Science Foundation
For current information write to:
Instructional Scientific Equipment Program
Division of Undergraduate Education in Science
National Science Foundation
Washington, D. C. 20550

United States Office of Education
Title VI of the Higher Education Act of 1965 is administered by the United States Office of Education, which offers support through matching-fund grants to institutions for the purchase of equipment to improve the quality of classroom instruction. It is pertinent to note that Title VI directs that considerations be given to enrollment pressures and the financial needs of the proposing institution.
This program has been developed in such a way that it involves both the U. S. Office of Education and the state governments. In general, the states set up their own priority and evaluation criteria, as well as the schedule for closing dates and awards. Final evaluation is made by the Office of Education and the grant is made directly by the Office of Education to the school.

Information on this program should be requested from the appropriate state agency that is responsible for higher education. This might be the State Department of Education, a Commission on Higher Education, or a comparable office. Requests made for information should include requests for copies of the pertinent information on criteria and on the format of the proposal itself. Information regarding the grant program may also be obtained from the following:

Chief, College Equipment Grant Section
Bureau of Higher Education
United States Office of Education
400 Maryland Avenue
Washington, D. C. 20202

The closing dates range from October 15 to February 15 in various states. Contact your local office for information on this date.

In order to take part in any of these programs, a proposal must be written by the principal investigator explaining what he needs, what he plans to do and how his proposal will benefit higher education.

The experiments in this manual will help to answer the question, "What can be done in nuclear chemistry?"

In writing this manual we are assuming that the reader has some familiarity with basic radiisotope measurements. In the General Appendix (Appendix I) at the end of the Manual we have included some valuable information in regard to licensing and Federal regulations for isotope use. A
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</table>

NOTES:
(1) Solid = solid; red = red; blue = liquid.
(2) Fusible = readily fusible.
(3) Values for gaseous elements are for liquid at the boiling point.

SARGENT-WELCH
SARGENT-WELCH SCIENTIFIC COMPANY
7300 LINDER AVENUE, SKOKIE, ILLINOIS 60076
CHICAGO • CINCINNATI • DETROIT • MINNEAPOLIS • PHILADELPHIA • HOUSTON
BOSTON • SPRINGFIELD, MASSACHUSETTS • SAN FRANCISCO • TULSA, OKLAHOMA
### TABLE OF PERIODIC PROPERTIES OF THE ELEMENTS

#### Percent Ionic Character of a Single Chemical Bond

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<th>Difference in Electronegativity</th>
<th>Percent Ionic Character</th>
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#### Sub-Atomic Particles

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<th>Neutron/Proton Number</th>
<th>Mass</th>
<th>Isotopes</th>
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### Notes:
1. For representative circles, higher valence of group. Circle is white, if color is red, if color is blue, and if color is purple. If both colors are shown, intensity of color indicates relative strength.
2. COVALENT RADIUS
3. ATOMIC RADIUS
4. ATOMIC VOLUME
5. THRESHOLD ENERGY (Threshold energy)
6. ELECTRONEGATIVITY
7. HEAT OF VAPOORIZATION (In cal/g-mole)
8. HEAT OF FUSION (In cal/g-mole)
9. ELECTRICAL CONDUCTIVITY (In ohm-1 cm-1)
10. THERMAL CONDUCTIVITY (In cal/cm-sec-1 cm-1)

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Catalog Number 1-18006

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