Physics 300 Laboratory Manual

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1. Proton Induced X-ray Emission Spectroscopy

a. Introduction and History

Proton Induced X-ray Emission (*PIXE*) Spectroscopy is an ion-beam analysis technique for multi-elemental analysis that can be used to determine the trace elemental composition and concentration of an unknown target material that has been bombarded by an incident particle beam. *PIXE* has been around since the early 1900's when Henry Moseley in 1913¹ discovered the effect, but the real utilization of it as an analysis technique occurred in the mid-1970's by the pioneers Johansson, Campbell and Malmqvist². The main idea behind *PIXE* is that there is a probability (called the ionization cross-section) that a moderately energetic particle, a proton in this case, can eject an electron from its orbit in an atom. After the ejection of this electron from the atom, an electron in a higher energy state will undergo a transition to this lower energy state and in the process emit a photon of a characteristic energy. This photon will be in the x-ray portion of the electromagnetic spectrum. The x-rays emitted from by each atom are characteristic to the atom. In other words, by knowing the energy of the emitted x-rays from an unknown target, we can fingerprint the elements in the target.

b. The Accelerator

The Ion Source

UC-IBAL's accelerator is a tandem electrostatic machine capable of reaching a terminal potential of *1.1MV* and can accelerate protons (hydrogen ions) and alpha particles (helium nuclei) to energies of *2.2MeV* and *3.3MeV* respectively. There are many other ion species that could be generated and accelerated, but our laboratory at present can only accelerate these two species. In order to perform a PIXE experiment, one must create and accelerate a species of particles, here protons.

To generate a proton beam one starts at the low energy end of the accelerator, as shown in Figure #1. The ion source is shielded from the operator by a cage and when the cage is removed, as in Figure #2, we can see the Alphatross Ion Source, and in particular the faint pink glow of the hydrogen plasma can be seen behind the fan on the right side. Hydrogen gas is bled from a storage bottle, not shown, in to a quartz bottle. Electromagnetic energy in the form of a radio frequency (RF) discharge is supplied to the hydrogen gas in the quartz bottle and this will produce positive ions. This energy strips the electron from the proton (a dissociation) in the hydrogen gas, and produces a plasma. If the process is left alone, the electron and the proton will recombine and will be subsequently restripped. The characteristic pink hydrogen discharge spectra can be seen in Figure #3, as well as two metal bands that couple the quartz bottle and the RF source. A voltage difference (usually about 2-6 kV) is used to push the ions out of the chamber through the exit aperture, making a continuous beam. To extract the protons into the accelerator, one uses a 2200 Volt bias voltage is placed across the ends of the bottle. The ends of the bottle are metal and the right end of the bottle is negative with respect to the left end of the bottle. This potential difference accelerates the proton out of the bottle towards the ion source.



Figure 1: Low energy end of the particle accelerator, showing the ion source under a cage. The cage is to protect the operator from a static discharge when the accelerator is operating.



Figure 2: Low energy end of the particle accelerator, showing the ion source with the cage removed. The ion source is operational as shown by the pink glow behind the fan on the right edge. The ion source holder can also be seen in the middle of the figure below the beam line.



Figure 3: Characteristic pink glow of a hydrogen plasma. The metal bands couple the RF source to the bottle and the bias voltage applied across the bottle extracts the protons into the accelerator, which is toward the right end of the photograph.

Here the electric field created in the bottle, due the bias voltage does work on the proton where, the work done is

$$W = q\Delta V_{bias} = (e)[2200V - 0V] = 2200eV \times \frac{1.6 \times 10^{-19}J}{1eV} = 3.52 \times 10^{-16}J.$$
 The work

done on the proton changes the proton's kinetic energy and hence its speed when the proton enters the ion source is approximately

W = $q\Delta V_{bias}$ = 3.52 × 10⁻¹⁶ J. = $\Delta KE \approx \frac{1}{2}m_p v_p^2 \rightarrow v_p$ = 6.5 × 10⁵ $\frac{m}{s}$, assuming that the proton starts from essentially at rest initially and the mass of the proton used was $1.67x10^{-27}kg$.

To produce a negative proton beam, the positive proton beam is immediately injected into a rubidium charge exchange cell. The ion source consists of a 10g mass of rubidium located in the furnace shown in figure #2. A furnace heats the rubidium and produces a low-density vapor of rubidium ions in the beamline. The protons pass through the rubidium (which have very loosely bound valence electrons) vapor and through charge-exchange collisions³, the protons gain two (2) extra electrons. This is shown in the schematic⁴ of figure #4.



Figure 4: Schematic of the ion source and charge exchanger for the positive to negative ion configuration.

The Pelletron Accelerator

The protons with their extra negative charges are now ready to enter the accelerator. The accelerator itself is a tandem electrostatic machine with a terminal potential of up to 1.1MV and further the ions are accelerated twice in succession. The Pelletron charging system charges the terminal by induction and the chain inside of the pressure tank consists of metal links connected by insulating links. The Pelletron charging system is shown schematically⁵ in Figure #5 and the actual inner workings of the Pelletron accelerator column are shown pictorially in Figure #6 and the chain and insulating links in Figure #7.



Figure 5: Pelletron charging system⁵, shown the chain and motor assembly. The chain motor drive system is located on the high-energy end of the accelerator. The terminal shell is located in the center of the pressure tube and the ions enter from the right hand side moving toward the left.

For a positive terminal Pelletron, which we have, the negatively charged inductor electrode pushes electrons off the pellets while they are in contact with the grounded drive pulley. Since the pellets are still inside the inductor field as they leave the pulley, they retain a net positive charge. The chain then transports this charge to the high-voltage terminal, where the reverse process occurs. When it reaches the terminal, the chain passes through a negatively biased suppressor electrode that prevents arcing as the pellets make contact with the terminal pulley. As the pellets leave the suppressor, charge flows smoothly onto the terminal pulley, giving the terminal a net positive charge. Most Pelletrons, as shown in the charging system diagram, employ "down-charging" as well as "up-charging." Down-charging works identically to up-charging, except the inductor/suppressor polarities are reversed, and it effectively doubles the charging current capacity of the chain. For the configuration shown here, small, slightly conductive "pickoff pulleys" provide the voltages for the terminal suppressor/inductor electrodes by drawing a tiny amount of charge from the chain, each pulley thus biasing the opposing electrode. This technique allows down charging "for free," i.e. without the use of high voltage supplies in the terminal. Depending on the particular design options, the system delivers charging currents of 100 - 200 µA or more per chain to the high voltage terminal. The drive pulleys, typically 30 cm to 60 cm in diameter, and motors are supported on movable platforms, which are counterweighted, automatically providing proper chain tension.



Figure 6: The accelerator column. The right end is the low-energy column, while the left end is the high-energy column. The metal tube in the center is the terminal. The protons enter the accelerator column at the low-energy end and are accelerated towards the high-energy end. Nitrogen gas bled in at the terminal strips the electrons producing again a positive beam of protons.



Figure 7: The Pelletron chain. The Pellets are the metal pieces the grey pieces in between each metal pellet are the insulating links.

The protons with their extra negative charges enter on the right end of Figure #6, called the low-energy end of the accelerator, since the ions here have a very low energy. They are accelerated through a 1.1MV potential difference and work is done on the charges. The work done is the work done to accelerate the single negative charge on the hydrogen ion is

 $W_{LE} = q\Delta V_{LE} = (-e)[0.0MV - 1.1MV] = 1.1MeV$. At the terminal, nitrogen gas is bled in from a bottle located on the high-energy end of the tank. The nitrogen gas (through charge exchange collisions) strips the electrons off of the proton, leaving only the positively charged proton. The proton now is accelerated through another 1.1MV potential difference away from the terminal, due to the repulsive force between the positively charged terminal shell and the positive charge of the proton. More work is done on the proton and the work done increases the energy of the charge and the proton leaves with a higher energy then when it entered the accelerator. The work done by the electric field accelerating the proton away from the terminal is

 $W_{HE} = q\Delta V_{HE} = (e)[1.1MV - 0.0MV] = 1.1MeV$. The total work done on the proton changes the proton's kinetic energy and hence its total change in kinetic

energy when the proton leaves the accelerator is given as the sum of all of the work that was done on the proton.

 $W_{net} = W_{bias} + W_{LE} + W_{HE} = 0.0022 MeV + 1.1 MeV + 1.1 MeV = 2.2022 MeV$. At this point, one should ask whether relativistic effects should be taken into account. The relativistic cutoff speed is $v \le 0.1c$ the effects of relativity to not have to be worried about. At this speed, we have a kinetic energy given by $KE = (\gamma - 1)m_pc^2$, where the proton's rest mass is approximately 938.3 $\frac{MeV}{c^2}$ and γ is the relativistic

correction to the kinetic energy given by $\frac{1}{\sqrt{1-\left(\frac{v}{c}\right)^2}}$. Evaluating the relativistic

kinetic energy at the cutoff speed gives us an energy threshold for relativistic effects. The cutoff energy, above which relativistic effects become important, is approximately 4.73MeV. Since we are well below this limit, we shall ignore relativistic effects and approximate the proton's speed by using the classical approximation to the kinetic energy, or $\frac{1}{2}m_pv^2$. Assuming that the proton starts essentially from rest at the ion source, and using the total done on the proton, 2.2022MeV, the proton's speed when it leaves the accelerator is approximately 2.1x10⁷m/s.

The Endstation

The accelerator endstation (or scattering chamber) consists of a stainless steel multi-way cross as shown in Figure #7 below. The ports around the circumference let the beam into and out of the scattering chamber as well as holds the x-ray detector and the feed-throughs for the solid state detectors (which are not installed at present, but in the future will be and further will be used to detect protons in a backscattering experiment.) In addition, there is a vacuum isolation valve and a turbo molecular pump that is used to create the vacuum in the scattering chamber. On the bottom of the scattering chamber are the mounts for the solid-state detectors; while on the top of the scattering chamber there is a 3axis target manipulator. The target manipulator is capable of moving targets vertically, horizontally left and right perpendicular to the beam, as well as horizontally back and forth parallel to the beam. The sample can also be rotated in the beam to change the angle of the beam with respect to the target normal. In Figure #7, the proton beam enters from the accelerator on the right side of the figure. A 3-sample target holder (not shown) is placed at the base of the target manipulator and any of three samples can be positioned in the proton beam. The beam passes through the sample and proceeds to the end of the beam line on the left hand side of the photograph. The end of the beam line is a Faraday cup (a device that collects charge), which is a connected to a charge integrator, so that the total charge incident on the target can be counted and will be used later in the analysis process.



Figure 7: Photograph of the accelerator endstation. The proton beam enters through the right end passes through the target in the center of the multi-way cross and exits out of the pipe on the left end. The 3-axis target manipulator is mounted on the top of the scattering chamber.

c. PIXE Theory

When a moderately energetic proton passes close by an atom, there is a probability, called the ionization cross-section for x-ray production, that the proton may eject an electron from the atom creating a vacancy. The *PIXE* process is shown in Figure $\#8^7$. Suppose that, as is shown in Figure #8, that the vacancy were created in the ground state of the atom (the n = 1 state of the Bohr atom), then the vacancy will be filled by an electron from any higher energy state. As a result of the electron's transition, an x-ray photon will be emitted. The energy of this x-ray is dependent of the atomic number of the atom and on the particular transition that the electron takes. This admits a very regular pattern of characteristic x-ray emission and was first discovered by Henry Moseley in 1913. This was the precursor to the materials analysis technique of *PIXE*, or proton induced x-ray emission spectroscopy. In this experiment, we use the accelerator to generate a beam of 2.2MeV protons and then make this proton beam incident on our target of interest. As the proton beam passes though the thin target material, x-rays characteristic to the elemental makeup target will be generated. Knowing the number of a particular species of x-ray detected allows for the determination of the concentration of that species of atom in the target and this is the *PIXE* method.

As is seen in Figure $\#9^8$, various x-ray transitions are shown for an arbitrary atom. Two things are needed to describe a specific transition, the higher and the lower energy states of the electron. From Figure #9, the letter (K, L, M, etc.) designations are used to describe the transition of an electron into a particular state. We call the ground state (the n = 1 state) the K-shell, the first excited state (the n = 2 state) the *L*-shell, and so forth. Electron transitions are possible from any higher energy state into a lower energy state, although not all transitions are equally probably. We designate the transition probabilities with Greek letters. For example, a transition of an electron from any higher energy state into the n =1 state is called a K-shell transition since the electron is going into the ground state, while a transition from any higher energy state into the n = 2 state is called an L-shell transition because the electron is transitioning into the first excited state. For any transition, say into the K-shell, the lowest energy, and therefore the highest probability, is termed an α -transition. A slightly more energetic transition, but less probable, is called a β -transition. Putting these two designating schemes together, we can label any transitioning electron from any higher energy state into the lower energy state where an electron had been ejected due to the passing by of the incident proton.



Figure 8: Diagram showing the PIXE Process of x-ray production by an incident proton of sufficient energy⁷.



Figure 9: Possible x-ray transitions for an arbitrary atom of nuclear charge Z^8 .

The energy of the emitted x-ray is defined as

$$\Delta E_{\rm x-ray} = E_{\rm upper \ state} - E_{\rm lower \ state} \tag{1}$$

where, the upper and lower states refer to the higher and lower energy states of the transition electron respectively. The energy of an electron in a particular state can be modeled reasonably well by using the Bohr theory⁹ of the atom, in which we assign the electron's energy as the sum of its kinetic energy (due to it's motion in the state) and potential energy (due to the separation of the electron and the nucleus.) Moseley working in Ernest Rutherford's lab, tabulated and compared the K_{α} frequencies (and hence the energies) of the characteristic x-rays emitted versus the atomic number of the atom, Z. He found that the frequency data were proportional to $(Z-1)^2$ due to shielding effects. When an electron is ejected from the K-shell due to interaction between it and the passing proton, the electron in the n = 2 state does not see the full complement of the nuclear charge since there is one electron left in the ground state. The effective nuclear charge that an electron in the n = 2 state will feel is the charge of the nucleus (Ze⁻) minus the charge of the electron (e^{-}) remaining in the ground state, and thus the energy of the transition is proportional to $(Z - I)^2$ using the Bohr Theory of the atom. Applying the Bohr theory to an atom of nuclear charge Z, and using expressions for the

kinetic
$$(K_n = \frac{m(Z-1)^2 e^4}{2(4\pi\varepsilon_0)^2 n^2 \hbar^2})$$
 and potential energies $(V_n = -\frac{m(Z-1)^2 e^4}{(4\pi\varepsilon_0)^2 n^2 \hbar^2})$, the

energy of an individual state is given by

$$E = -\left(\frac{me^4}{2(4\pi\varepsilon_0)^2\hbar^2}\right) \frac{(Z-1)^2}{n^2} = -13.57eV\left(\frac{(Z-1)^2}{n^2}\right),\tag{2}$$

where we have the mass of the electron, $m = 9.11 \times 10^{-31} kg$, its charge

 $(e^{-} = 1.6 \times 10^{-19} C)$, $\varepsilon_o = 8.85 \times 10^{-12} \frac{C^2}{Nm^2}$, $\hbar = \frac{h}{2\pi} = 1.055 \times 10^{-34} Js$, and we've converted from Joules to electron-volts using the conversion factor $1eV = 1.69 \times 10^{-19} J$. The negative sign in equation #2 is a reflection of the fact that the electron is in a bound state around its parent nucleus. Thus equation #1 may be written as

$$\Delta E_{x-ray} = E_{upper \ state} - E_{lower \ state} = -13.57 eV \cdot (Z-1)^2 \left[\frac{1}{n_{lower}^2} - \frac{1}{n_{upper}^2} \right].$$
(3)

Equation #3 is the relationship that Moseley found in 1913 and plots of the atomic number versus the square root of the energy (since the energy is proportional to $((Z-I)^2)$ are called Moseley plots, shown in Figure #10⁹, and they show the regions over which we expect to see K-shell, L-shell, etc. production of x-rays by an energetic incident proton. Thus light elements we would expect to see K-shell x-ray production and for heavier elements we would expect the x-rays to be produced from transitions into the L-shell (mainly because our beam energy is not sufficiently large to eject the tightly bound inner shell electrons of heavy Ztargets.) In theory one uses equation #3 to calculate the charge of the nucleus with which the incident proton interacted. This involves knowing n_{upper} and n_{lower} based on the x-ray spectra obtained and a cursory knowledge of the type of elements (light or heavy) that you might have in your target and the energy of the emitted x-ray from the spectra. In practice, one does not do this. Calibrating GUPIX with standards allows one to identify the elements, based on x-ray tables (stored in GUPIX) that were generated using equation #3. Thus, in practice, we look up what the elements are from calibrated x-ray spectra and you tell GUPIX which elements to look for when you perform the analysis.



Figure 10: An example of a Moseley plot showing the typical ranges for K- and L-shell x-ray production by an incident proton of sufficient energy.

d. PIXE Experiment

The *PIXE* experiment that we will be performing will be to determine the elemental composition and concentration of several unknown atmospheric aerosol samples. In order to determine the trace elemental composition and eventually the concentrations of the trace elements found, we will need to calibrate our detector system and analysis software GUPIX. To calibrate our detector system and our analysis software a set of single element standards with known concentrations will need to be analyzed first. We will run three single element standards and then we will run two atmospheric aerosol samples. After each run, we'll name the standards using the following convention: Elemental Standard The Date Charge Collected Lab Group.mca. For the atmospheric aeorslols, we'll use the same naming convention, except we'll change the file name from Experimental Standard to AA Number. After all of your experimental runs, you will need to find your saved files. Open each of them up with a textediting program. You will see in each file a set of information in the header and footer. Delete the information in the footer and in the header. In the header only, on the very first line, you want to put in the following: "1024 0" without the quotes. The analysis software will eventually use this information and we want to tell the software how many channels of data there are in the file (1024 channels) and to start numbering the channels with channel zero. Save your file with the same name, but change the file extension to ".dat", again without the quotes. Programs like Microsoft Excel can open these data files^{\circ}. If you use Excel then you need to import the data. You should include the PIXE graph in your lab write-up, as this is your data.

GUPIX is the *PIXE* analysis software of choice and it was (and still is) developed by John Campbell and his research team at the University of Guelph in Guelph, Ontario, Canada⁶. *GUPIX* will be installed on the computers in S&E N303 for your use during the term. To determine the elemental composition and concentrations of the elements found in the target, we have to set-up GUPIX. Open the GUPIX program on the desktop and under file, open *New Project*. Navigate to where your files are located and open one of the standards. GUPIX will display something like spectrum seen in Figure #11 below. Now we will need to set the analysis parameters in GUPIX.



Figure 11: Typical elemental spectrum generated by GUPIX from an experimental PIXE run.

The following recipe will be used to determine the trace elemental composition and concentrations of the elements in your target.

• Under the *Setup Menu*: choose *Trace* from *Trace or Matrix* and then select *Trace Element in a Known Matrix* as shown in Figure #12.



• Under the *Setup Menu*: choose *Setup*. This will bring up a window where you can select the parameters in the initial setup as shown in Figure #13 and input your parameters.

Aperimental setup Options	<u>a</u>
Angles	Detector Parameters
Beam normal 0 X+ay normal 45	Select a Detector O-Union SDD Edit Detector File Help
Eas Peandes Inn (PEDTAL _ Energ Bet) (200 Inn (PEDTAL _ Eas Bet) (Our restin	Discore Option Option

Figure 13: Experimental parameters setup window.

- Under the Sample Menu choose Sample Structure then choose Thin
- Under the *Sample Menu* choose *Trace Element Solution* and then *Define Fit Elements* as seen in Figure #14. Add the elements that you believe are in your sample.

Select	all ele	ement	s to b	e fitte	d in ti	he spe	ctrun	1										
' H			Per	riod	ic Ta	able	of t	he E	lem	ent	s						He	Z Atomic #
° li	Be											° B	ໍເ	'N	0	°F	¹⁰ Ne	
Na	12 Mg											13 A I	¹⁴ Si	¹⁶ P	¹⁰ S	¹⁷ Cl	18 Ar	
¹⁹ K	20 Ca	21 Sc	Ti	23 V	24 (r	25 Mn	²⁸ Fe	27 Co	28 Ni	29 Cu	³⁰ Zn	Ga 31	32 Ge	33 As	³⁴ Se	38 Br	^{≫8} Kr	
37 Rb	°" Sr	³⁹ Y	Žr	41 Nb	42 Mo	₄≎ Tc	⁴⁴ Ru	*8 Rh	** Pd	47 Ag	*" Cd	⁴⁰ In	ŝ	Sp 21	52 Te	50 	×4 Xe	
°° Cs	se Ba	67 La	⁷² Hf	73 Ta	74 W	76 Re	76 Os	77 r	78 Pt	79 Au	∞ Hg	°1 TI	⁸² Pb	83 Bi	⁸⁴ Po	∞ At	^{⊗5} Rn	
⁸⁷ Fr	® Ra	⁸⁹ Ac																Special Elements
		se Ce	^{so} Pr	Nd	Pm	⁶² Sm	е» Еи	°4 Gd	°° Tb	°° Dy	67 Ho	⁶⁸ Er	^{co} Tm	⁷⁰ Үb	71 Lu]		□ 102 □ 103 □ 104 □ 105
		°° Th	Pa	92 U											-			□ 106 □ 107 □ 109 □ 109
							01		C	ancel								E 110

Figure 14: Define Fit elements window. Click on all the elements that you believe are in your sample.

• From the *Fit Menu* choose *Spectrum Details* and fill in the parameters of your fit range and of the energy equations. You will need to enter parameters for the values of A_1 , A_2 , $A_3 = 0$, A_4 and A_5 . To generate the parameters for A_1 and A_2 you will need to make an energy calibration plot. Using the data given below, construct a plot of the *channel number versus the energy* (in *keV*) for the data given in the table #1 below. Curve fit and use the curve fit to determine A_1 , the y intercept and A_2 the slope of your plot. For the coefficients A_4 and A_5 use 4.025 and 0.617 respectively. In addition, select the *Digital Background Filter* box and just say ok to the choices that you are given.

Channel Number	Energy (keV)
349	9.441
409	11.069
439	11.887
514	13.992
653	17.747
764	20.781
967	26.345

Table 1: Data of channel number and x-ray energy generated by using radioactive 214 Am calibration source on the Union SDD detector.

• Lastly, from the GUPIX Menu choose RUN. You can ignore the warning messages that GUPIX gives you. If everything runs correctly you should see a box like Figure #15.



Figure 15: The Output of the GUPIX engine. The upper window gives the GUPIX fit to the data and the lower window gives the Residuals.

The concentrations C_Z of each of the elements Z are given by the following equation:

$$C_{Z} = \frac{Y_{Z}}{Y_{T} \cdot H \cdot Q \cdot \varepsilon \cdot T}$$
(4)

This is how GUPIX does its calculations and in equation #4 we have that Y_Z is the intensity of the principle x-ray line for element Z, Y_T is the theoretical intensity,

from a set of standards, H is an experimental constant (equal to the solid angle of the detector) that needs to be determined from a set of standards, Q is the total charge collected, ε is the efficiency of the detector, and T is the transmission coefficient through any filters or absorbers between the sample and the x-ray detector. In the parameter set above, we had to specify Q and any absorbers that we had, so that *GUPIX* knows the values of Q and T. The efficiency is included in the parameter file when you selected the *Union SDD* detector and the Y_T are included in *GUPIX*'s database. After GUPIX engine runs and you generate the graph above, click view stats button in the upper right hand corner. This will generate a set of stats on the spectra that you just ran. This is shown in figure # 16 below.



Figure 16: Statistics generated by GUPIX. The concentrations are given as an areal density in ng/cm^2 .

The only parameter we do not know is H, the experimental constant. To determine H, we first assign it a value, one. Then, by running on the standards, we can determine the exact value since we know both the concentration determined by GUPIX and also the exact concentration listed on the standard. Taking the ratio of the concentration given by GUPIX to the actual value listed on the sample will determine H. Select "do another fit" from the File menu and under the Set-up menu enter the new value of H that you determined in the experimental options box. Rerun GUPIX and see if the concentration of the element in the standard matches the value given on the slide. Repeat this procedure for each of your standards and make a plot of *H* vs. *Z* for each of the standards. Is H roughly a constant? Should it be? (Yes it should be. H is analogous to what's called the solid angle of the detector (or the ratio of the area of the detector to surface area of a sphere centered on the target and ending at the location of the detector. Since none of these distances changes during the experimental run, H should be constant. However, it may fluctuate here since you are only doing three standards and not say 30 standards, where H would be more constant. Take an average value for H for your experimental runs.)

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2. Mössbauer Spectroscopy of ⁵⁷Fe

a. Introduction/Background Information

Mössbauer spectroscopy is a method for measuring small shifts in nuclear energy levels with high precision. This method involves the "recoilless" emission and absorption of gamma rays and utilizes the Doppler effect in order to use these gamma rays as a sensitive probe. Measurements of small shifts in nuclear energy levels provide information about the electromagnetic environment of the nucleus and the structure of the material in which it resides.

Resonant Absorption

A quantum system can undergo a transition when it absorbs or emits a photon of a specific energy. The energy of the photon is dependent upon the difference in the energy levels of the transition. The energy of the ground state is absolute, but the energy of the excited state is not a precisely defined quantity. Due to the Uncertainty Principle, the natural line width is given by

$$\Gamma = \frac{\hbar}{2\tau},\tag{1}$$

where Γ is the natural line width of the excited state and τ is the lifetime of the state.



Figure 1: Illustration of the emission of a gamma ray photon from a nuclear transition and the corresponding recoil of the nucleus.

However, when a photon is emitted by a free system the photon energy is not equal to the transition energy. This is because the system recoils due to conservation of momentum, as shown in figure #1, and takes with it some of the transition energy in the form of kinetic energy. If we call the energy of the transition E_T and the recoil energy E_R , then the energy of the emitted photon can be expressed as

$$E_{\gamma} = E_T - E_R. \tag{2}$$

We can rewrite this expression in terms of the recoil momentum p_R as

$$E_{\gamma} = E_T - \frac{p_R^2}{2m}.$$
 (3)

Due to conservation of momentum since the nucleus is assumed to be initially at rest, we see that

$$p_R = p_\gamma = \frac{E_\gamma}{c},\tag{4}$$

where p_{γ} is the momentum of the photon. By combining Equations 3 and 4 we find that the energy of the emitted photon is

$$E_{\gamma} = E_T - \frac{E_{\gamma}^2}{2mc^2}.$$
 (5)

Likewise, when the photon is absorbed the absorbing system recoils. Therefore, the distributions of the emission and absorption energies are separated by twice the recoil energy. The probability of resonant absorption is proportional to the overlap of these distributions. In atomic systems this probability is high because the recoil energy is small compared to the natural line widths. In nuclear systems, however, the recoil energy is much larger than the natural line widths, and therefore the probability of resonant absorption is very small.

Recoilless Emission and Absorption

In 1958, Rudolf Mössbauer showed that for atoms bound in a lattice, a nucleus doesn't recoil individually. The recoil momentum, therefore, is taken up by the entire lattice, which has a very large mass. From equation (5) we see that as $m \rightarrow \infty$, $E_{\gamma} \rightarrow E_T$. Therefore, when the nucleus is embedded in a massive substrate the recoil energy is negligible. This concept applies to absorption of photons as well.

Doppler Shift

When there is relative motion between the emitter and the absorber there is a Doppler shift in photon energy. The energy of the photon is given by the Lorentz transformation

$$E'_{\gamma} = \frac{1}{\sqrt{1-\beta^2}} \left(E_{\gamma} + v p_{\gamma} \right) = E_{\gamma} \left(\frac{1+\beta}{\sqrt{1-\beta^2}} \right), \tag{6}$$

where $\beta = \frac{v}{c}$, E_{γ} and p_{γ} are the energy and momentum of the emitted photon, v is the relative velocity between the emitter and the absorber, and E_{γ} is the resulting Doppler shifted photon energy. For $\beta << 1$ we take the first order of the binomial expansion to get

$$\Delta E = E'_{\gamma} - E_{\gamma} = \beta E_{\gamma} = \frac{v}{c} E_{\gamma}$$
(7)

as an expression for the change in photon energy due to the motion.

By varying the relative velocity between the emitter and absorber, a range of photon energies can be scanned. In Mössbauer spectroscopy we measure the absorption rate as a function of velocity, which can be converted to energy shift. Analysis of the absorption spectrum yields information about the electromagnetic environment of the nucleus.

Use of ⁵⁷Fe in Mossbauer Spectroscopy

In our Mössbauer spectroscopy experiment we use ⁵⁷Fe. Our γ -ray source is ⁵⁷Co, which decays to ⁵⁷Fe via electron capture as seen in Figure #2. When the iron decays from the I=3/2 to the I=1/2 state a *14.4-keV* gamma ray is emitted, which is the photon of interest. For absorbers we use foils of stainless steel and natural iron.



Figure 2: A schematic diagram of the decay scheme of ⁵⁷Co. The cobalt undergoes electron capture, yielding an electron neutrino and a ⁵⁷Fe atom in an excited state. The iron undergoes a number of nuclear transitions, one of which yields a 14.4-keV gamma ray. This is the gamma ray of interest.

The resolution of the spectrometer is characterized by the ratio $\frac{\Delta E}{E}$. The first excited state of ⁵⁷Fe, has a natural line width $\Gamma = 2.4 \times 10^{-9} eV$, and the transition produces a photon of *14.4-keV*. Thus, the ideal resolution of our apparatus is *1.7x10⁻¹³*. The actual resolution of the spectrometer is approximately the width of the absorption peak of stainless steel.

Isomer Shift

If the chemical environment of the iron nuclei in the source and in the absorber is different, then the electron densities around the nuclei will be different. The electromagnetic interaction between the electrons and the nucleus depends on the electron density, so if the host materials are different there will be a shift in the resonance energy from the source to the absorber, which can be seen in Figure #2.

We see this effect when we take a Mössbauer spectrum of stainless steel. The iron nuclei created in the cobalt have a different host material than the iron nuclei in the stainless steel. This causes a shift in the absorption spectrum of the stainless steel.



Figure 3: A schematic diagram of the isomer shift due to differences in the chemical environments of the emitter and absorber.

Nuclear Zeeman Effect

The nucleus of an iron atom in natural iron and certain other iron compounds is in a strong magnetic field caused by the electrons of the atom and of neighboring atoms. Since both of the nuclear energy levels of the 14.4-keV transition have spin and associated magnetic moments, they experience hyperfine splitting caused by the interaction of the magnetic moment of the nucleus with the magnetic field, as shown in Figure #4. These new levels are the magnetic substates of the energy levels, which are degenerate in the absence of a magnetic field, and this results in more possible nuclear transitions. The transitions that are allowed are determined by the quantum selection rule, which states that $\Delta m_I = 0, \pm 1$.



Figure 4: An illustration of the isomer shift and nuclear Zeeman effect in ⁵⁷Fe

The change in energy caused by the interaction of a magnetic moment with a magnetic field can be expressed as

$$\Delta E = \vec{\mu} \cdot B \tag{8}$$

where μ is the magnetic moment of the state and *B* is the strength of the magnetic field. This expression holds for any system. When quantum mechanics are applied, this expression can be rewritten as

$$\Delta E = -g\mu_N B_z \left(\frac{m_I}{I}\right) \tag{9}$$

where g is the g-factor or gyromagnetic ratio, $\mu_N = \frac{e\hbar}{2m_p}\mu_N$ is the nuclear magneton, B_z is

the component of the magnetic field \vec{B} in the direction of the magnetic moment $\vec{\mu}$, m_I is the magnetic substate, and I is the spin of the state. This expression gives us the shift in energy due to the nuclear Zeeman effect. The energy of the resulting transitions is

$$E = \left(E_e + \Delta E_e\right) - \left(E_g + \Delta E_g\right) \tag{10}$$

where E_e and E_g are the energies of the excited and ground states, and ΔE_e and ΔE_g are the shifts in the energy levels due to the nuclear Zeeman effect. The expression can be rewritten as

$$E = \left(E_e - E_g\right) + \left(\Delta E_e - \Delta E_g\right) \tag{11}$$

If we set

$$E_0 = \left(E_e - E_g\right) \tag{12}$$

and

$$\Delta E_{transition} = \left(\Delta E_e - \Delta E_g\right) \tag{13}$$

the expression becomes

$$E = E_0 + \Delta E_{transition} \tag{14}$$

where E_0 is the energy of the transition if the levels were not split and $\Delta E_{transition}$ is the energy shift caused by the nuclear Zeeman Effect.

The Mossbauer Spectrometer

A schematic of the Mössbauer spectrometer is shown in Figure #5. The source is attached to a linear motor that sweeps through a range of velocities at constant acceleration. The motor is controlled with the S-700A Mössbauer drive circuit. The absorber is positioned between the source and a krypton gas proportional counter that counts the photons that are transmitted through the absorber and measures their energy. The pulses from the detector are amplified and analyzed with the MCS-pci card in the computer. A software gate is placed around the *14.4-keV* peak in the photon energy spectrum and the number of *14.4-keV* photons is plotted as a function of source velocity. When the Doppler shifted gamma rays have energy that corresponds to a nuclear transition energy, the photons will be absorbed and then re-emitted in all directions. At these velocities we will see a decrease in the number of gamma-ray counts in the detector. The correlation between the source velocity and the number of photons is controlled by the MCS-pci card and the velocity and drive signals between the linear motor and the Mössbauer drive circuit are monitored on an oscilloscope.



Figure 5: A schematic diagram of the Mössbauer spectrometer.

b. Pre-Lab Exercises

Do the following pre-lab exercises and hand them in separately from your lab report.

Consider the measurement of hyperfine splitting of the first excited and ground states of ⁵⁷Fe with a Mössbauer spectrometer.

- 1. Derive an expression for the measured velocity of a transition, $v_{m_{I_g} \to m_{I_e}}$, as a function of I_e , I_g , m_{Ie} , m_{Ig} , g_e , g_g , μ_N , B_z , E_γ , and c.
- 2. Use your general expression for $v_{m_{I_g} \to m_{I_e}}$ to write explicit expressions for each of the transition velocities $v_{-\frac{1}{2} \to -\frac{3}{2}}$, $v_{-\frac{1}{2} \to -\frac{1}{2}}$, ...
- 3. Use the expression for the transition velocities to derive an expression for the gyromagnetic ratio of the excited, g_e , in terms of the gyromagnetic ratio of the ground state, g_g , and the ratio of differences in transition velocities.

c. Procedure for the Mossbauer Spectroscopy Experiment

This section explains how to perform the experiment. There are important notes at the end of each section, so please read them carefully. A diagram of the experimental setup is shown in Figure #5.

Equipment: Setting Up the Experiment

Assemble the following equipment:

- NIM bin
- Mössbauer spectrometer track with collimator holder, absorber holder, and detector holder
- ASA K-4 Mössbauer Linear Motor
- ASA S-700A Mössbauer Drive Module
- Mössbauer absorbers (a-iron, natural iron, and stainless steel)
- GE Reuter-Stokes RS-P3-1605-262 X-Ray Proportional Counter
- Ortec 142PC Preamplifier
- Ortec 556 High Voltage Power Supply
- Ortec 572A Amplifier
- PC with Ortec MCS-pci Multi-Channel Scaler and MCS-32 software
- Tektronix TDS 340A Oscilloscope

Connections

Make the following connections using BNC, SHV, and MHV cables (see Fig. #5):

- Linear motor DRIVE BNC → S-700A rear DRIVE BNC → Oscilloscope channel 2
- Linear motor VELOCITY BNC → S-700A rear VELOCITY BNC → Oscilloscope channel 1
- Preamp power connector \rightarrow Amplifier rear preamp power connector
- Proportional counter MHV \rightarrow Preamp INPUT MHV
- High voltage power supply SHV \rightarrow Preamp BIAS SHV
- Preamp ENERGY BNC \rightarrow Amplifier IN BNC
- Amplifier UNI output BNC \rightarrow MCS-pci SCA IN BNC
- S-700A rear DATA OUT BNC → MCS-pci START IN BNC
- MCS-pci MIDPASS OUT BNC → S-700A rear BISTABLE BNC → Oscilloscope external trigger

Settings

Turn on the oscilloscope, the NIM bin, and the high voltage power supply (*HVPS*). Then apply the following settings to the electronics modules:

- High voltage power supply \rightarrow +1800 V
- Amplifier: Gain $\rightarrow 0.5$, Course Gain $\rightarrow 100$, Shaping Time $\rightarrow 0.5$
- S-700A Drive Module: Velocity thumb wheel → 100 (±10.0 mm/s), Mode Switch → C.A. ASYM., FIDELITY → ~ 10 o'clock
- Oscilloscope: Ch1 \rightarrow 2 V, Ch2 \rightarrow 200 mV, M \rightarrow 50 ms

Calibrations

We will use the well-known absorption peaks for α -Fe to calibrate the velocity scale of the spectrometer.

- Collimate the α -Fe absorber and attach it to the absorber holder.
- Place the absorber holder on the spectrometer track so that the absorber is directly in front of the hole in the plate in front of the detector.
- Attach the ⁵⁷Co source to the linear motor and slide the motor towards the detector so that the source slides into the collimator holder and the collimator holder is against the absorber holder.
- Build a shielding wall around the spectrometer with the lead bricks.
- Start the MCS-32 program and select SCA Sweep under the Acquire menu.
- Stop the data acquisition after you have enough data to see a good 14.4-keV peak. Use the mouse to select the region around the 14.4-keV peak as shown in Figure #6 and click the Set SCA button on the SCA Sweep dialog box.
- Select Input Control from the Acquire menu and click on the Use SCA Input button on the Input Control dialog box as shown in Figure #7.
- Select Pass Control from the Acquire menu and set up the dialog box as shown in Figure #8.
- Select Time Units from the Display menu.
- Start the data acquisition and then turn the S-700A Drive Module Run switch ON. Monitor the velocity waveform on the oscilloscope (Ch2). After a short time, the velocity waveform should settle down into a regular sawtooth pattern and the LEDs on the drive module should blink alternately. If this condition is not established within a few minutes, adjustments will have to be made to the drive module (see the documentation for the S-700A Mössbauer Drive Module).
- When you have a good α -Fe spectrum with six well-defined absorption peaks like that shown in Figure #9, stop the data acquisition.
- The well-known absorption peaks for a-Fe irradiated with a ⁵⁷Co(Rh) source occur at velocities of -5.418, -3.182, -0.946, 0.734, 2.970, and 5.206 mm/s. We will use these values to calibrate the velocity axis of the spectrometer. Position the cursor at the minimum of the left-most absorption peak and select Calibrate from the Calculate menu. The Calibration Worksheet shown in Figure #10 will appear. Type -5.418 into the value window of the Calibration Worksheet and click Enter. Move the cursor to the minimum of the next peak, type the corresponding velocity into the Value window, and click Enter. Repeat this process for the other four absorption peaks. Check the accompanying graph to make sure that the calibration function provides a reasonable fit to the data. If so, click the Apply button to apply the calibration function. Close the Calibration Worksheet and unselect Time Units in the Display menu to display the spectrum in velocity units.
- Take Mössbauer spectra for both stainless steel and natural iron absorbers.



Figure 6: A $\gamma\text{-ray}$ energy spectrum from the ^{57}Co source with the 14.4-keV peak of interest highlighted.

Input Cor	itrol 🛛 🖓 🔀
	SCA Lower: 3.1616 Volts
Use SCA Input	Upper: 4.1187 Volts
	0.0000 9.9976 Discriminator
Use Disc Input	Rising Edge © C Falling Edge 50 0hm © © 1k 0hm
	-1.6V 3.0V
	OK Cancel

Figure 7: The Input Control dialog box.

Pass Length	Trigger	
1000 ÷ 4 · 65535 Channels	Internal	
Pass Count Preset	Acq Mode G Sum C Replace C Rep/Sum	
Internal C External Bin Width: 200.00000 us V	OK Cancel	

Figure 8: The Pass Control dialog box.



Figure 9: Mössbauer spectrum of α -Fe.

Data Po	pint	Polynomial	Apply	
Time: 0.1662	µs ⊂ ⊂ s	Linear 🗸		
/alue: 5.2060		Zero Intercept Zero Slope	Restore	
Enter		Zero Quadratic 🗖	Close	
6 Data Poin	ts	Show Graph 🖌	^	
Channel	Value	Delta (%)		
175.00	-5.418	-5.7572	1	
328.00	-3.182	-1.8516		
470.00	0.946	+1.6004 -1 //E//0	~	

Figure 10: The Calibration Worksheet dialog box.

Finishing Up

- Set the voltage on the HVPS to 0V. When the voltage runs down turn off the HVPS.
- Turn off the NIM bin and the oscilloscope.
- Return the ⁵⁷Co source to its lead capsule and return this to its lead housing.

Cautionary Notes

- **THE** ⁵⁷**Co SOURCE IS RADIOACTIVE**. Always be careful when working with or handling it. Keep it away from your body while handling it, and make sure to use the lead bricks to create a wall between the source and yourself while performing the experiment.
- The window of the detector is very sensitive and will break easily. **DO NOT** touch it, and be careful when working near it.

d. Data Analysis

Extract the isomer shift and the width of the absorption peak in energy for the stainless steel absorber.

From the spectrum on natural iron, extract the isomer shift, the g-factor of the first excited state given the g-factor of the ground state (0.09044 ± 0.00007), and the value of the magnetic field strength at the nucleus.

e. References

For more information see the following references:

- 1. Krane, Kenneth S., "Introductory Nuclear Physics," Wiley (1987).
- 2. Melissinos, Adrian C. and Jim Napolitano, "Experiments in Modern Physics," Second Edition, Academic Press (2003).
- 3. "Mössbauer Effect: Selected Reprints," American Institute of Physics.

3. Environmental Radioactivity

a. Background and Theory

Throughout the course of any given day, one is subjected to sources of radioactivity, primarily natural radioactivity, but occasionally anthropogenic sources are encountered. In this set of experiments we will be looking at some sources of natural radioactivity by detecting the gamma ray emissions from unstable atoms. In particular in this study of environmental radioactivity, we'll be identifying several sources of natural radioactivity using a gamma ray detector and from data taken on several of these sources, calculate the half-life the sources. Radioactive isotopes have half-lives that range on the order from seconds or less up to billions of years. This of course, depends on the particular radioactive isotope you are studying. Of particular interest to physicists are the decay processes that have long half-lives, which are thus useful in determining the ages of geological samples that can be used to determine the age of the Earth, or by studying meteorites the age of the solar system.

In order to determine the age of a sample of material from the environment, a particular species of radioactive isotope needs to be studied. In particular, the activity (number of decays of a particular unstable atom per unit time) of the sample of radioactive isotopes being studied needs to be determined. The activity is measured in units of the *Curie* or the *Becquerel*, where the conversion $1Ci = 3.7 \times 10^{10} Bq$ and $1Bq = \frac{1\text{decay}}{s}$ is the standard unit of radioactivity. From the activity one can determine the decay constant associated with the particular unstable, or radioactive atom. The decay constant is related to the probability of a decay of an unstable atom per unit time and will be ultimately related to a quantity called the half-life of the sample, or the time over which it takes the activity of the sample to decay by a factor of two.

Any unstable (or radioactive) atom will undergo a decay process in which the parent atom in an excited or high-energy state will transform itself into a daughter atom (of lower energy) with an emission of particles or light. Radioactive atoms are able to decay to a final stable end state in several distinct ways by usually through a decay chain of multiple emissions of particles or light. These decays are labeled on an energy level diagram like the one shown in figure 1.



Figure 1: Energy level diagram for the decay of a generic atom of mass 137. This diagram shows the basic decays possible and the probability of decay. In addition the half-lives are also given for a specific decay.

In Figure 1 above, for a mass 137 nucleus, the vertical axis shows the massenergy in units of keV while the horizontal axis shows the atomic number Z, or the number of protons in the nucleus, which increases to the right. Diagonal arrows represent the ways in which the nucleus can decay into a lower energy state as well as the probability of a particular occurrence of decay. Half-lives are also given for a particular type of decay.

These forms of decay products that can be emitted from the radioactive nucleus are alpha decay (the emission of a helium nucleus from the decaying atom), beta-plus and beta-minus decay (the emission of a positron or electron from the decaying nucleus) or gamma ray emission. Each of the decays are mathematically shown below, where in each decay, the parent atom is symbolized by ${}^{A}_{Z}X$ and the decay product ${}^{A'}_{Z}Y$. For a particular decay, both energy and charge are conserved and each parent atom is assumed to be at rest before the transmutation (conversion of the parent into a daughter atom) and subsequent energy release.

1. Alpha decay:

$${}^{A}_{Z}X \rightarrow {}^{A-4}_{Z-2}Y + {}^{4}_{2}He \tag{1},$$

where the alpha particle is ejected at a particular speed that depends on the particular decay involved. The daughter atom recoils to conserve momentum in the system. For example, americium-241 ($^{241}_{95}Am$) is an alpha emitter with alpha

particles being emitted with energies XXX (x% of the time) and YYY (y% of the time.)

2. Beta-plus decay:

$${}^{A}_{Z}X \rightarrow {}^{A}_{Z-1}Y + {}^{0}_{+1}e + v_{e}$$

$$\tag{2}$$

where an unstable neutron decays into a proton and an electron neutrino. A classic example of beta-plus decay is the decay of sodium-22 $\binom{22}{11}Na$.

3. Beta-minus decay:

$${}^{A}_{Z}X \rightarrow {}^{A}_{Z+1}Y + {}^{0}_{-1}e + \overline{v}_{e}$$
(3)

where an unstable proton decays into a neutron and an anti-electron neutrino. An example of beta-minus decay is the decay of cesium-137 $\binom{137}{55}Cs$ into barium-137 $\binom{137}{56}Ba$). A small fraction (8%) of the time the beta-minus particle carries away all of the excess energy, and 92% of the time the resultant barium-137 is left in an excited state which decays to the ground state with an emission of a 0.662MeV gamma ray. Thus the rate of gamma ray production is 92% of the source activity.

4. Gamma decay:

$${}^{A}_{Z}X^{*} \rightarrow {}^{A}_{Z}X + {}^{0}_{0}\gamma \qquad (4),$$

where the "star" on the parent state represents a metastable (or rather long-lived excited nuclear state) by the emission of a high energy electromagnetic wave. The non-starred state is the lower energy state. An example is the decay of barium-137 as mentioned above.

In this experiment we will study several types of radioactive atoms that decay to final end states by the emission of gamma rays. We will learn how to use and calibrate a gamma ray detector as well as take data on the decay of ${}^{137}_{56}Ba$ to determine its half-life. Then we will use these techniques and apply them to the gamma decay of an unknown source and use the spectra generated to determine the identity and the half-life of this source.

b. Ge(Li) Gamma Ray Detector

Ge(Li) stands for a lithium drifted germanium detector. This detector is a type of solid-state detector made out of a crystal of germanium with the impurity sites doped with lithium. A schematic of our detector, a Canberra model GC1017, is shown in Figure 2 below.

Model 7500 Vertical Dipstick Cryostat



Figure 2: Schematic of the Canberra Model 7500 series gamma ray detector. Our particular detector, the GC1017 Ge(Li) is virtually identical.







Figure 4: Schematic of the Multi-Channel Analyzer assembly. The detector signals from the pre-amplifier are fed into an amplifier and then converted from analog to digital signals and sent to the MCA and displayed on the computer screen.

The gamma ray detector is a Canberra Model GC1017 that is cooled by liquid nitrogen in order to reduce electronic noise due to the vibration energy in the germanium crystal. A fraction of gamma rays that are incident from a radioactive source placed on the detector actually interact with the detector, many pass on through. When a gamma ray does interact with the detector it will ionize electrons in the germanium giving up its energy to the electron it liberates. This electron will in turn ionize more germanium atoms producing many electrons. If a strong electric field (controlled by a high-voltage power supply connected to the crystal and set to 3500V) is put across the crystal the electrons can be collected and counted as shown in Figure 3 above. The number of electrons counted is a measure of the incident gamma ray's energy. The signals heights that are detected are in general small and need to be analyzed by a pre-amplifier.

The signals from the pre-amplifier are sent to an analyzer and then to a multichannel analyzer (MCA) that is operated in pulse-height or PHA mode as shown in Figure 4 above. In PHA mode, the input pulses are sorted into channels according to their amplitude. The MCA provides a visual display of the resulting distributions and s all modern MCA's are microprocessor-controlled and have fairly extensive local arithmetic operation capabilities. In this experiment, the MCA will be used in PHA mode to investigate nuclear γ -ray spectra. The PHA measures the height of an incident pulse and special circuitry converts this incident signal into a voltage peak and from the maximum height of the pulse (as say would be read off of a oscilloscope) is assigned a channel number based on its height. The amplitude of an incoming analog pulse (normally in the 0-10 V range) is digitized by an analog-todigital converter (ADC) and the resulting digital value is used as the address of a memory location corresponding to a given channel. Thus the screen display of the number of counts vs. channel number is really a histogram of the number of counts vs. pulse height, i.e. a pulse height spectrum. The range of pulse heights to be analyzed can be set via upper and lower level discriminators at the input and are preset by the operator.

c. Energy Calibration of the Ge(Li) Detector

In order to perform any type of qualitative analysis of gamma ray spectra using the Ge(Li), we need to calibrate our analysis software. The software that we will be using for this experiment is called MaestroTM and is marketed by Ortec.

To calibrate the detector software, we'll use several known radioactive sources. The sources we will use are all sealed sources of fairly low activity. These sources are ${}^{22}_{11}Na$, ${}^{57}_{27}Co$, ${}^{137}_{55}Cs$, and ${}^{133}_{56}Ba$. Wearing gloves, carefully and quickly take each source out of its container and place it on the center of the gamma ray detector and take spectra. In addition, be sure to remove the previous source (if one was present) and putting it back in its appropriate container. Take spectra (noting the real time data was acquired, the live time that the detector was actually recording data, and the dead time) for each of these sources and save the spectra with your name, the source name, and the date taken. From the spectra, record the channel number and energy

associated with each of the photopeaks, as shown by example in Figure 5. The energies of each of the decays are found in the *Table of Isotopes* book found in the lab.

In Excel or similar program, prepare graphs for each of your calibration spectra and prepare a graph of the energy of each photopeak versus the channel number for each photopeak of your calibration sources. Does the data approximate a straight line? We usually assume that the energy scale is linear in the channel number. What is your calibration equation? For each of your calibration spectra, change the horizontal axis from channel number to energy. Do the energies of each of the photopeaks correspond to the energy from book of the *Table of Isotopes*, located in the lab, according to your calibration curve?



Figure 5: Example of the gamma ray emission spectrum of an unknown radioactive sample.

d. Determination of the half-life of ${}^{137}_{56}Ba$.

The ${}^{137}_{56}Ba$ that we will use for this experiment is a metastable excited state of a nucleus left behind when the ${}^{137}_{55}Cs$ emits a beta-minus particle. The ${}^{137}_{56}Ba$ is the daughter of the parent nucleus ${}^{137}_{55}Cs$. The gamma ray emitted has an energy of 0.662MeV and is emitted when the excited state barium nucleus decays to its ground state as is exhibited by equation (4). Both the cesium and the barium are radioactive. The barium has a short half-life while the cesium has a long half-life. The barium will be separated from the cesium by using a mild acid solution and this process is called milking. In the cow (shown in figure 6) there is powdered cesium in a mesh filter. When the cesium decays to barium, the barium is trapped in the filter. The acid solution can be analyzed using MaestroTM.



Figure 6: Cs/Ba isotope generator kit on the left. The cow is shown on the right with the syringe of the acid solution attached. The cow is milked into a pan for use in an experiment from Pasco. http://www.pasco.com/prodCatalog/SN/SN-7995_isotope-generator-kit-barium-137-m/#overviewTab

Since barium has such a short half-life you want to have a plan in place before milking the experiment. This plan has to include selecting a region of interest to count over and to make sure MaestroTM is set up correctly. To get ready but before you start, you are going to have to choose a region of interest to count over. You will have to milk the cow into the metal pan and place it on the top of the gamma ray detector. DO NOT DO THIS YET! GET THE FOLLOWING STEPS IN MIND FIRST BEFORE YOU PROCEEDE. As soon as you place the metal pan with the barium solution on the detector press the start button on the Maestro[™] toolbar and count for about 10 seconds and then press the stop button on the toolbar. Using the cursor highlight the region where the counts are above the background and right-click with the mouse and choose Mark ROI (or region of interest.) Clear the spectra and under the Sevices/Job Control menu, select JOB control and choose the program called *Decay.JOB*, and choose open. This program called Decay.JOB will start to run and it will take data for you. When the program is opened the program will start to run and you don't have to do anything at this moment except watch the spectra build up. The contents of the program *Decay.JOB* are shown below. This program is designed to be automated and allow you to take multiple spectra really quickly (or really slowly depending on the parameters you've set) and save each individual run. This eliminates you having to start/stop the program manually.

SET_DETECTOR 3 SET_PRESET_CLEAR SET_PRESET_LIVE 15 LOOP 150 CLEAR START WAIT FILL_BUFFER SET_DETECTOR 0 REPORT "DECAY???.RPT" SET_DETECTOR 3 END LOOP This program is a while loop. The loop will run a preset number of times as long as a set of parameters is satisfied. The first line, SET_DETECTOR 3 instructs the software which detector to use. Here the Canberra detector is detector 3. Don't change this, as the experiment will not count anything. The SET_PRESET_CLEAR clears any preset instructions that you might have had. SET_PRESET_LIVE 15 command instructs the software to count for a live time of 15 seconds and you can change this to anything that you want. Here I'd leave it set at 15 seconds. The LOOP 150 command repeats the command loop 150 times and you too could change this but I'd again leave it for the moment. The next three commands execute the loop. CLEAR clears the memory; START starts the experiment counting for 15 seconds. After 15 seconds, the software stops writes the file to DECAY???.RPT, where "???" is the number of the file, 001 for file #1, 002 for file #2, etc and then the software waits for 1 second and then executes the loop again until it reaches 150 times.

Once you are confident in the steps involved, you are ready to begin the experiment. Milk the cow into one of the metal dishes using about 1mL of the acid solution. Make sure that you have the cow and syringe setup in the correct orientation as labeled on the cow. Place this metal dish with the barium solution on the center of the Ge(Li) detector and take data for 10 seconds, select a ROI to count over. Clear the spectra and then start the *Decay.JOB* program.

When you have finished, open the following path: My Computer:

c://programfiles/Maestro/. Save all of your report files on a thumb drive and delete the files in this file so that the next person's data doesn't get ruined. When you have saved all of your files, open each and record the gross counts and determine the time that each of these gross counts corresponds to. This will take some thinking to get the time axis correct. Next determine your activity, the number of gross counts per 15-second interval. Open Excel and put your data of activity and time into two columns. Prepare a graph of the activity as a function of time. What is the shape of your data curve? To determine the functional form of the data, make three graphs. For one, plot the data on a Cartesian Plot, for the second, a semi-log plot, and the third a loglog plot. Which one gives you a straight line? If the Cartesian plot, put a straight-line curve fit through the data. If it is a semi-log plot, put an exponential curve-fit through the data.

The activity of the sample is changing with time and is related to the probability of decay (which is called the decay constant, λ which is unknown) and the number of radioactive atoms at time t = 0, also unknown. The relationship is given by

$$\frac{dN}{dt} = -\lambda N_0. \tag{5},$$

where, $A_0 = \lambda N_0$ is defined as the activity at time t = 0.

Solve equation (5) for the theoretical relationship for how the number of radioactive atoms of the sample changes as a function of time. Make sure you state your initial conditions in your solution. From your graph and the relationship determined from equation (5) what is the decay constant λ ?

The half-life is defined as the amount of time it takes for the activity of the sample to decrease by a factor of two. Derive a relationship for the half-life of ${}^{137}_{56}Ba$ and then determine the half-life in an appropriate choice of units. What is the half-life of ${}^{137}_{56}Ba$ with its uncertainty? How does your measured value compare to the actual value? What are the sources of this uncertainty?

e. The Radioactive Decay of $^{40}_{19}K$

For this part of the experiment we will determine the half-life of $\frac{40}{19}K$, a radioactive isotope of potassium that occurs naturally at an abundance of about 0.0118%. The sample we will use is ordinary potassium chloride salt, *KCl*, which is not classified as a radioactive substance, and therefore is perfectly safe to handle without special precautions. In fact, you may have seen a product in the grocery store sold under the name "*Lite Salt*", which contains a mixture of *KCl* and ordinary table salt. Even so, there are enough disintegrations of $\frac{40}{19}K$ that we can easily measure and count the rate of radioactive decay using the Canberra GC1017 gamma ray detector.

One of the radioactive decay products of ${}^{40}_{19}K$ is ${}^{40}_{18}Ar$, and is of particular interest to geophysicists because of its utility in determining the age of potassium bearing rocks, and therefore allowing us to make estimates of the age of the earth.

Before you start taking data, you need to do a couple of things. The first is to calibrate your energy spectrum so that you know where to look for the decay of ${}^{40}_{19}K$. To do this, take your last spectrum using ${}^{133}_{56}Ba$. Under the *Calculate* menu, select *Calibrate* and then *destroy calibration*. Next, use the cursor to select an appropriate region of interest around the ${}^{133}_{56}Ba$ peak and then mark this region. Right click on your region and select *Peak Info*. This will give you the channel number of the peak. Then under the *Calculate* menu, select *calibrate* to do a one-point calibration. The channel number should already be entered and you need to tell it what the energy is at this channel number. Enter in the energy of this ${}^{133}_{56}Ba$ peak in *keV*. If the channel number is not automatically inserted, insert it in the appropriate spot. Now, look up the decay energy of ${}^{40}_{19}K$.

To start data taking, weight the sample holder and then weigh out $(0.70 \pm 0.01)g$ of *KCl*. Spread the *KCl* evenly over the entire bottom of the sample holder, which will be a piece of aluminum foil. Place the sample holder on the gamma ray detector take data for a few minuets to locate the ${}^{40}_{19}K$ peak. Stop the data collection and mark an appropriate region of interest for the ${}^{40}_{19}K$ peak. Clear the spectra and under the *Services* menu, select *JOB* control and choose the program called *Decay K.JOB*, and

choose open. This program will count the decay events for 10 1-minute periods record the results. Save your data on a thumb drive using the weight as part of your filename.

Weigh out another 0.3g of *KCl* and add to the sample holder (for a total now of 1.0g). Place this on the gamma ray detector and start the *Decay_K.JOB* program again and count for 10 1-minute periods and record the results.

Add another 1.0g of *KCl* and repeat the counting process, using the *Decay_K.JOB* program again and count for 10 1-minute periods and record the results.

Repeat this one more time by adding a final 1.0g of *KCl* and repeat the process again. You should have data sets for four different masses, 0.7g, 1.0g, 2.0g, & 3.0g of *KCl*. Take one last spectrum of the background with no sample of *KCl* near the detector. Take this spectrum for at least 10 minutes and determine the average count rate per minute for the $\frac{40}{19}K$ background.

Now, calculate the average counts per minute for the 10 one-minute counting periods for each mass of *KCl*. Subtract the average background counts per minute to get net ${}^{40}_{19}K$ counts per minute. Divide by the mass of *KCl* for that particular sample run to get <u>counts</u> *KCl*.

The reason you had to do several sample runs with varying amounts of *KCl* is due to the fact that as the sample mass increases, the depth of the *KCl* layer in the sample holder increases. The particles emitted by decaying ${}^{40}_{19}K$ nuclei on the top of the heap of *KCl* are partly absorbed by other *KCl* particles lower in the sample, so counts do not increase linearly with mass of *KCl*, but instead begin to level off at some point. Make a plot of counts per minute versus mass of *KCl* and be sure to use the point(0,0) since if there was no *KCl* then there should be no counts detected (in the absence of the background of course).

Next make a plot of the net $\frac{counts}{\min g}$ KCl versus the mass of the sample of KCl. Since radioactive decay products are partly reabsorbed by the KCl sample itself, and thus never reach the detector, we need to figure out what the counts would have been had there been no reabsorption at all, which could only happen with a layer of zero thickness. Therefore, the y-intercept of the above plot gives you a way to determine the counts per minute per gram at zero grams of KCl, or zero thickness, of KCl. Determine the $\frac{counts}{\min g}$ for zero thickness, of KCl.

Now, we need to worry about the efficiency of the gamma-ray detector. We neglected this in the study of barium. (A good idea? Maybe. Maybe not.) Thinking about how the detector and sample are arranged the photon resulting from

any given decay event may be produced in any direction, but only those that are produced towards the detector will be counted. The detector efficiency (is a function of energy and ideally should be calculated – but that's for another lab experiment) and for this detector, it is approximately 2%. In other words, only about 2 in 100 events actually get counted. Actually since the detector's efficiency is energy dependant, this is about 2% for 1400*keV* gamma rays. At lower energies, the detector is more efficient but that's not where our peaks are. We therefore we have to calculate a corrected value for the $\frac{counts}{\min g}$. Using your value for the $\frac{counts}{\min g}$ for zero thickness, of *KCl*, divide this by the detector efficiency and the value you calculate by doing this is the corrected value for the $\frac{counts}{\min g}$ if the detector was 100% efficient. This is the activity of the sample per gram of $\frac{40}{19}K$.

Now, determine the number of ${}^{40}_{19}K$ atoms in 1 gram of potassium using the molecular mass of *KCl* (don't forget that not all of the potassium in the sample is radioactive.) Using the value of the activity the sample per gram of ${}^{40}_{19}K$ and the number of ${}^{40}_{19}K$ atoms per gram in the sample, determine the decay constant for ${}^{40}_{19}K$. Lastly determine the half-life of ${}^{40}_{19}K$ and compare this along with its uncertainty to the actual value of the half-life of ${}^{40}_{19}K$.

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