

# X-ray Fluorescence and Moseley's Law

## 1 Background

### 1.1 Ordering of the periodic table

The 19<sup>th</sup> century saw many efforts to arrange the elements in a sensible order. The chemist John Dalton prepared one of the first tables of the elements in 1803, ordering them by increasing atomic weight. In the following decades it was recognized that groups of elements like lithium, sodium and potassium shared similar chemical properties, suggesting the presence of recurring patterns of chemical behavior. Using this concept, Dmitri Mendeleev developed around 1870 what is now known as the periodic table. Elements were placed in order in the periodic table, largely according to their atomic weight, and numbered consecutively, although no physical meaning was attached to these “atomic numbers.” But this scheme had some puzzling anomalies, one of which was the ordering of nickel and cobalt; cobalt has higher atomic weight (58.93) than nickel (58.69), but its chemical properties suggest that it should precede nickel in the periodic table. This anomaly and others (atomic weight of argon is greater than that of potassium, same for tellurium and iodine) were gradually leading to the realization that the significant parameter in terms of ordering the periodic table was not atomic weight but rather the number of electrons and therefore the number of positive charges in the atom. A definitive resolution of these anomalies would await the X-ray measurements of H. G. Moseley.

In 1897 J. J. Thomson discovered the electron and established its discrete nature. The atomic model built on this discovery (the so-called raisin-pudding model) was one with positive charge distributed continuously throughout the atom with the electrons located at equilibrium positions in the atom, about which they oscillated when perturbed. Ernest Rutherford's alpha particle scattering measurements put this model to rest, as they demonstrated that the positively charged part of the atom consisted of a small, dense core, later termed the nucleus. In 1913 Niels Bohr, working in Rutherford's laboratory in Manchester, England, proposed a theory of the atom which combined Rutherford's results with the concept of quantized radiation developed by Planck and Einstein. In Bohr's model, the electron moves about the nucleus in a circular orbit, the attractive force resulting from the Coulomb interaction between the negatively charged electron and a nucleus with equal and opposite charge. The key breakthrough in Bohr's model was the quantization of electron angular momentum. Using this model, Bohr was able to derive the formula for wavelength of the light for the various transitions (also known as lines) in hydrogen-like spectra,

$$\frac{1}{\lambda} = R \left[ \frac{1}{n_f^2} - \frac{1}{n_i^2} \right] Z^2. \quad (1)$$

The integers  $n_i$  and  $n_f$  represent the angular momentum (units of  $\hbar$ ) of the initial and final states of the electron, and  $Z$  is an integer, known as the atomic number, representing the units of charge in the nucleus.  $R$  is the Rydberg constant;  $R_\infty = me^4/4\pi c\hbar^3$ , where  $m$  and  $e$  are the electron mass and charge, respectively, with the subscript denoting that this formula applies to an infinitely heavy nucleus. For a nucleus of finite mass  $M$ , the electron's mass  $m$  must be replaced by its reduced mass,  $mM/(m + M)$ .

Not only did the Bohr model accurately describe the spectrum of the hydrogen atom where  $Z = 1$ , but also for the singly ionized helium atom where one electron orbits a nucleus with  $Z = 2$ . The

success of the Bohr formula with the hydrogen and helium atoms suggested that it may also apply to transitions in heavier elements. If this proved to be the case, one would be able to order such transitions as a function of  $Z$ , thus providing an unambiguous means of ordering the elements in the periodic table.

The stage is now set for Moseley's measurements. It was already known that atoms of a given element emitted X-rays at well defined, characteristic energies. Armed with the new Bohr model, Moseley set about to measure the characteristic wavenumbers,  $1/\lambda$ , (inverse wavelengths, also proportional to frequency) for a number of different elements, and to correlate the X-ray frequency for a given element with the atomic number,  $Z$ , of that element.

Moseley's measurements were spectacularly successful. In the words of Robert Millikan (of Millikan oil drop/electron charge fame):

“In a research which is destined to rank as one of the dozen most brilliant in conception, skillful in execution, and illuminating in results in the history of science, a young man twenty-six years old threw open the windows through which we could glimpse the sub-atomic world with a definiteness and certainty never dreamed of before.”

Moseley's life was cut tragically short—he was killed at age 27 fighting for the British in World War I.

By measuring the wavenumber associated with a particular line (now designated K), from the spectrum of each element, Moseley established that the lines from a large number of elements obeyed the relation

$$\frac{1}{\lambda} = C(Z - \sigma)^2. \quad (2)$$

This relation is now known as Moseley's Law.  $C$  and  $\sigma$  are constants with  $C \propto R$  (Rydberg constant) and  $\sigma \approx 1$ .  $\sigma$  is known as the screening constant. The connection between this formula and the Bohr formula is that  $1/\lambda \propto Z^2$  in both cases. A plot representing Moseley's data is shown in Fig. 1. The data can be interpreted as resulting from an electron transitioning from an initial state with  $n = 2$  to a final state with  $n = 1$ , with one other electron screening the nucleus, thus reducing the effective nuclear charge to  $Z \approx 1$ . An understanding of why the nucleus is screened by only one electron for the K lines would await the development of quantum mechanics in the 1920's.

Moseley's work provided an unambiguous justification for ordering the elements in the periodic table according to atomic number  $Z$ , rather than Mendeleev's original ordering by atomic weight  $A$ . His work helped resolve several outstanding problems with the table as it had existed: the above mentioned anomalies with heavier elements preceding lighter elements; the assignment of  $Z = 44$  to ruthenium, implying that the element  $Z = 43$  (technetium, which has no stable isotopes and must be synthesized in a nuclear reactor or particle accelerator) was as yet undiscovered; and the prediction of other as yet undiscovered elements at  $Z = 61, 72$  and  $75$  [1].

With each element emitting X-rays at unique wavenumbers, X-ray analysis has become a powerful tool for analyzing chemical composition. X-ray analysis is used to determine the elements present in biological, environmental and geological samples. Figure 2 shows the X-ray spectrum from a water pollution sample, with channel number on the x-axis being proportional to X-ray energy, and the y axis showing the intensity of the detected X-rays. An X-ray detector very similar to the one you will use in this experiment was used on the Mars Sojourner Rover to analyze the composition of Martian rocks and soil. A spectrum obtained on this mission is available in the manufacturer's literature.

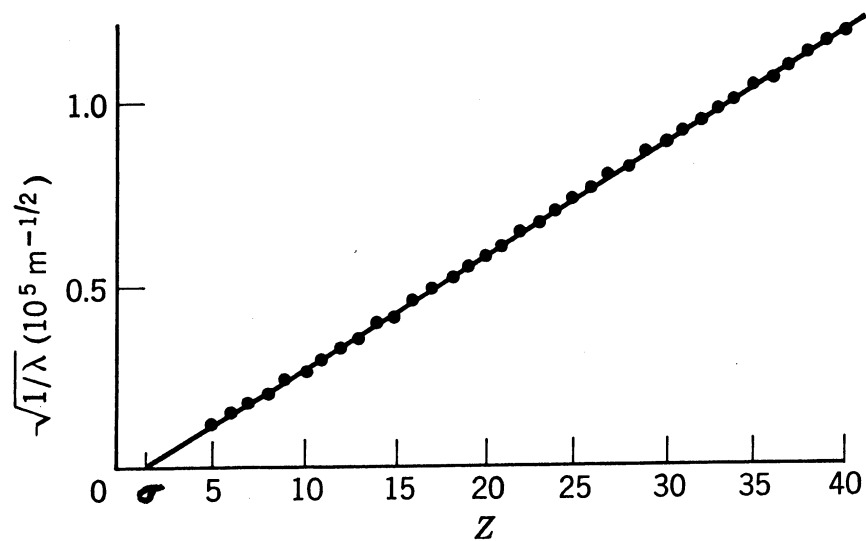


Figure 1: Moseley's data. The line represents a fit to data using Moseley's Law.

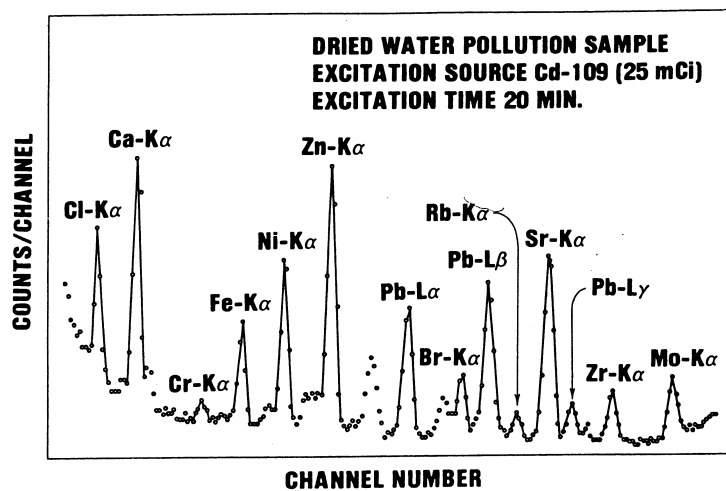


Figure 2: X-ray spectrum of a water pollution sample.

As an aside, X-ray energy is obtained from wavenumber  $1/\lambda$  by multiplication with  $hc$  where  $h$  is Planck's constant and  $c$  is the speed of light; remember  $E = \text{energy} = h\nu$  (Greek “nu”) and  $\nu = c/\lambda$ .

## 1.2 Designations of the various X-rays

Moseley selected out one particular line, or energy, from the spectrum of each element to make the plot in Fig. 1. This is to say that there are more than one X-ray line associated with each element. This can be understood in terms of electrons transitioning between the various inner shells within the atom. Each shell is designated by a quantum number  $n$ , and an associated letter;  $n = 1$ , the innermost shell, is known as the K shell,  $n = 2$  is known as the L shell,  $n = 3$  is known as the M shell, and so on. Fig. 3 shows the possibilities for various transitions (vertical scale not linear – closer to logarithmic). The open arrows pointing upward represent electron transitions from the  $n = 1$ ,  $n = 2$ , etc. levels to the cross-hatched region known as the continuum, where electrons are no longer bound to the atom. Such a transition results in a vacancy in the electron shell structure. The vacancy is filled when an electron from an higher lying shell (higher means greater  $n$ ) transitions to a lower lying shell, accompanied by the emission of a characteristic X-ray. All transitions to the K shell are known as K lines, with the transition from the L shell known as the  $K_\alpha$  line, from the M shell as the  $K_\beta$  line, and so on. Transitions to the L shell are known as L lines, and transitions to the M shell are known as M lines. The K lines are clearly more energetic than the L lines, and the K lines more energetic than the M lines.

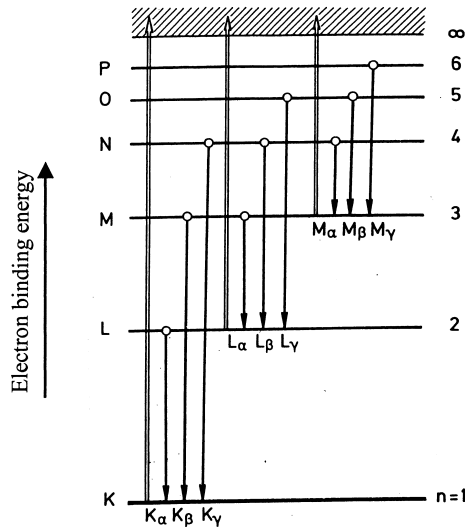


Figure 3: Diagram of K, L, M, etc. shell energy levels and identification of the various X-ray lines.

Within a given series of lines, K for example, the  $\alpha$  line will clearly have less energy than the  $\beta$  line, and the  $\beta$  line less energy than the  $\gamma$  line. Many of the lines have sub-levels resulting from spin-orbit coupling and from screening effects [2]. These various levels are denoted by numerical subscripts, e.g.  $K_{\alpha 1}$ ,  $K_{\alpha 2}$ . The energy differences between these sublevels are quite small for the lower  $Z$  elements. For iron ( $Z = 26$ ) the  $K_{\alpha 1}$  and  $K_{\alpha 2}$  energies are 6.403 and 6.390 keV, respectively. This energy difference is too small to be resolved by our detector. At higher  $Z$ , the energy differences become appreciable; for lead, the  $K_{\alpha 1}$  and  $K_{\alpha 2}$  energies are 74.957 and 72.794 keV, respectively.

This splitting into sublevels is known as the “fine structure” of X-ray spectra.

Usually the  $K_\alpha$  line is the most prominent feature of an X-ray spectrum. The  $K_\beta$  line will typically have 10-15% of the intensity of the K line for  $Z \leq 30$ , with the intensity ratio increasing to about 30% for the higher  $Z$  elements. A spectrum showing the  $K_\alpha$  and  $K_\beta$  lines for a manganese target is shown in Fig. 4. For lower  $Z$  elements, identifying the K lines is usually the easiest way to determine the composition of a target; the L lines for these elements have very little energy and may not be detected efficiently by the detector, if detected at all. At higher X-ray energies the detector efficiency drops off so much (see attached efficiency curve for our detector) that the K lines for the higher  $Z$  elements will be hard to see. In this case, it may be easier to identify a high  $Z$  target by looking for its (lower energy) L lines, which are more efficiently measured by the detector.

### 1.3 How X-rays are generated

X-rays are photons with energy in the range of approximately 1 keV to 100 keV with corresponding wavelengths of approximately 1 nm to .01 nm. As mentioned previously, an X-ray results when an electron transitions between inner shells of an atom. For such a transition to occur, there must be a vacancy in one of the inner shells to which an electron can transition. How is such a vacancy created? There are three different ways: bombardment of the target with fast electrons; irradiation of the target with highly energetic photons, and by radioactive decay. In the first two cases, the particle striking the target must have sufficient energy to overcome the binding energy of the ejected electron. With electron bombardment, a sufficiently energetic electron striking the target can eject an inner shell electron from a target atom, thus creating the vacancy; this is how X-rays are generated in medical and dental X-ray machines. In the second case, an inner shell electron is also ejected, but this time by a sufficiently energetic photon. In the third case, a “parent” nucleus changes from  $Z$  to  $Z - 1$  by capturing an electron from the innermost (K) shell, thus leaving a vacancy in the electron shell structure of the “daughter” atom. (This mode of radioactive decay is known as electron capture.)

In our apparatus, X-rays are produced by bombarding a target with energetic photons, namely, gamma rays. These photons result from the radioactive decay of  $^{57}\text{Co}$  to  $^{57}\text{Fe}$  by electron capture. After the decay, the daughter  $^{57}\text{Fe}$  nucleus is left in an excited state, and it decays to the ground state by emitting photons with energy of 122 keV and 14 keV (and sometimes a single photon with energy 136 keV). Note the usage: photons which originate in the nucleus are called “gamma rays” even if their energy is the same as those arising from transitions in the electron shell (“X-rays”), as is the 14 keV mentioned. The  $^{57}\text{Fe}$  atom also emits an X-ray as the K shell vacancy in the  $^{57}\text{Fe}$  atom is filled. You may see these peaks in your spectra as well as the characteristic peaks coming from your sample.

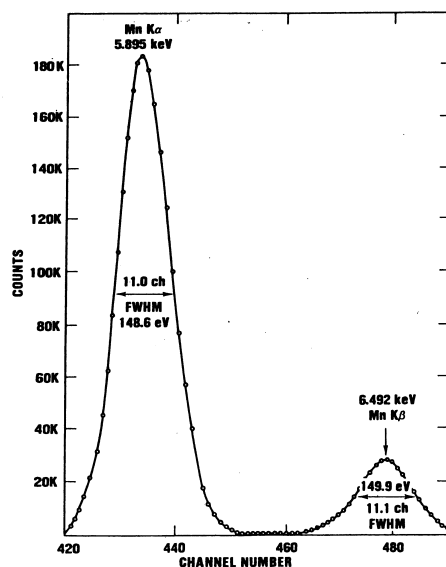


Figure 4: X-ray spectrum from a manganese target.

## 2 The experiment

**Experimental Goal 1:** Demonstrate a version of Moseley’s Law with a detector that converts photon energy to peak voltage of an electronic pulse by studying a set of known samples. Establish the relationship given in Eq. (2), for which the peak voltage  $V$  (expressed in units called *channel numbers*) is proportional to  $1/\lambda$ . Use these measurements to determine the screening constant  $\sigma$  for the K x-rays.

**Experimental Goal 2:** Determine the  $Z$ , and subsequently the element name, of six “unknown” samples using two different analysis methods:

**Method 1:** From your Moseley’s Law trend, match the peak voltages directly to  $Z$ .

**Method 2:** Via a calibration and look-up table. To do this, use the known samples. For a subset of the known samples, look up the known x-ray energies for the various peaks. Then create a plot of measured peak voltages (in channel number units) versus known x-ray energies (in keV). Make a fit to this (it should be a line). Use the fit result to create a “calibration curve.” The calibration curve function should allow you to enter a channel number and obtain an energy in keV. Test your calibration with other known energy/channel-number pairs. Then feed in the measured voltages from the unknowns to determine measured energies. Use the measured energies and the look-up table to establish the elements. (This second method is the modern approach to spectroscopy.)

In this experiment, we bombard elemental samples with gamma radiation from a radioactive source to induce x-ray fluorescence. The x-rays emitted by the sample are detected by a solid state radiation detector, which produces electronic pulses whose amplitudes are proportional to the energy of the absorbed x-rays. Digitization electronics that perform “pulse height analysis” are used to record the pulses and histogram their amplitudes (in channel numbers) for subsequent analysis.

The known samples, in order of increasing  $Z$ , are listed in Table 1. Before starting, run the apparatus for 10–30 minutes without a target to use as a comparison for runs with a target in

Symbol	$Z$
Cl	17
Ti	22
Cr	24
Fe	26
Ni	28
Cu	29
Zn	30
Ag	47
Cd	48
Sn	50
Gd	64
Ta	73
Au	79
Pb	82

Table 1: Table of known samples used in this lab. Note: The Cl source is in the form NaCl. Na ( $Z = 11$ ) has too low of an x-ray energy to be measured with our equipment.

place. You want to be able to see the peaks which constitute the “background.”

Measure spectra from all of the known samples, following the advice given in Section 3.5.

Identify any peaks above the background and locate their centers. For low- $Z$  samples, you should be able to locate  $K_\alpha$  and  $K_\beta$ . For higher  $Z$ , you may see a split in the  $\alpha$  lines to obtain  $K_{\alpha 1}$  and  $K_{\alpha 2}$ . With these and higher  $Z$  still, you will start to see the L lines.

Make a table that organizes the results, listing the element and the positions of the lines. Try to group each series in a single column (or row).

Also acquire X-ray spectra for the 6 unknown targets. Use your results above to make an initial determination of  $Z$  of each unknown target from the tabulated results. Create a similar table for the unknowns. Your estimate of  $Z$  for these is likely to change.

Plot all of the peak positions from the knowns in a “Moseley plot”:  $\sqrt{V}$  vs.  $Z$ , where  $V$  (given in units of channel numbers) is the voltage corresponding to the peak.  $V$  is proportional to the energy of the X-ray. You should see that the positions lie recognizably along lines corresponding to the different x-ray series.

Next, identify the  $K_\alpha$  series from as many of the known samples as possible and fit the  $\sqrt{V}$  vs.  $Z$  for this series to a line. Determine the constants  $C'$  and  $\sigma$ , along with their fit uncertainties, in the equation

$$\sqrt{V} = C'(Z - \sigma) . \quad (3)$$

Repeat the above for the (smaller) set of L lines to obtain the screening constant for this series.

Regarding the unknowns, for method 1, plot the peak positions of the peaks from the unknowns on the same graph as the knowns, and if necessary, adjust the value of  $Z$  in order to make the peaks lie nicely within the pattern established for the knowns for acquired spectrum and your  $\sqrt{V}$  vs.  $Z$  trend.

The equations (2) and (3) are almost the same. The peak voltage is proportional to the energy, which is proportional to  $1/\lambda$ , but there is no predefined relationship between  $V$  and  $1/\lambda$ . Thus, even

though we expect  $C$  to be proportional to  $C'$ , we do not know *a priori* what the proportionality constant is because  $V$  depends on the detector design and the detector's associated electronics (bias, amplification, time constant, etc.).

Because of this, our experiment cannot independently determine a value for  $C$  in Eq. (2). However, one can obtain an estimate for the screening constant  $\sigma$  easily. It is a pure number equal to the x-axis intercept (assuming that the x-axis is the atomic number  $Z$ ). For what  $C$  should be, solve the following exercise.

**Exercise 1** *Bohr's model predicts that the Rydberg constant is given by  $R_\infty = me^4/4\pi\hbar^3 = 1.0974 \times 10^7 \text{ m}^{-1}$ , where  $m$  and  $e$  are the mass and charge of the electron, respectively. To compare a measurement to Bohr's theory and  $R$ , use the known fact that for iron ( $\text{Fe}$ ,  $Z = 26$ ) the  $K_{\alpha 1}$  x-ray has an energy of 6.403 keV to estimate a value for  $C$  in Eq. (2). How does this compare to that predicted by Bohr's equation, Eq. (1)?*

Moseley's law is an approximation that works well for the K series of x-rays, but not so well for the L and other lines.

In addition, there may be an offset in the conversion between the voltage and the energy of your detector: the use of a single point at 6.403 keV is not enough to establish a good calibration for your detector.

Thus "Method 2," which takes care of the problems of using Moseley's law alone at the cost of not having a single theoretical function to predict the element. In this method, make a plot of known energies, from the look-up table, versus measured voltages. Fit this to a line so that you can convert any voltage (i.e., channel number) to an energy (in keV). This is a "calibration curve" for your detector. Use this calibration to convert the measured peaks of the unknowns to measured energies.

Also for method 2, include the peaks in the higher- $Z$  materials that represent the L-lines. These lines are visible in the samples for  $Z = 47$  (Ag) and above. Apply your calibration and see if the energies agree reasonably with the look-up table.

From the calibrated energy measurements and look-up table, check your initial determination of  $Z$  for the unknowns. You may find some puzzling results. In these cases, look again at the raw data sets, and see whether the unknown spectrum resembles ones with K-lines at the energies you are interested in or L-lines at those energies. Remember: one peak energy by itself is often not enough to identify the element! Also pay attention to what is reasonable, for example, your sample is unlikely to be a rare element (like plutonium) or a gas (like xenon)! Simply looking at the sample will give you some hints.

### Extension Ideas

1. In most cases where the  $K_\alpha$  line is strong, one can easily see the  $K_\beta$  line at slightly higher energy. This should also follow Moseley's law pretty well. Compare the fit parameters to Eq. (3) for these series to each other and to the Bohr equation prediction.
2. Take a much longer background run (1-2 hours or longer) and try to identify the X-rays in the background which are superimposed on any data you obtain with a given target. (Note: could some peaks come from the Co-57 source itself?)
3. Use Moseley's Law to determine the primary elemental composition of samples of your choice.



4. Develop a multi-element sample with 3 to 4 strong peaks across the energy range that can be used to calibrate the energy scale in a single measurement.

## 3 Apparatus and Procedure

### 3.1 General Comments

This apparatus consists of a  $^{57}\text{Co}$  radioactive source (same source as in the Mössbauer Experiment), a set of targets of different elements, and an AmpTek XR-100CR semiconductor detector. This detector measures the X-ray energy directly, in contrast to Moseley's method of measuring X-ray wavelength. The advantage of a detector like ours is the compact size and ease of operation. However, for resolving closely spaced lines in an X-ray spectrum, wavelength measurement remains the standard.

The XR-100CR detector is optimized to detect photons in the energy range of 1 to 30 keV. The efficiency of the detector drops off rapidly at higher energies (see Fig 5). The output of the XR-100CR detector is conditioned and amplified, and then sent to a pulse height analyzer (PHA). This device plots number of counts on the y axis vs. voltage, in the form of channel number or voltage, on the x axis. A typical plot of the PHA output will look something like the plots in Figs. 2 and 4.

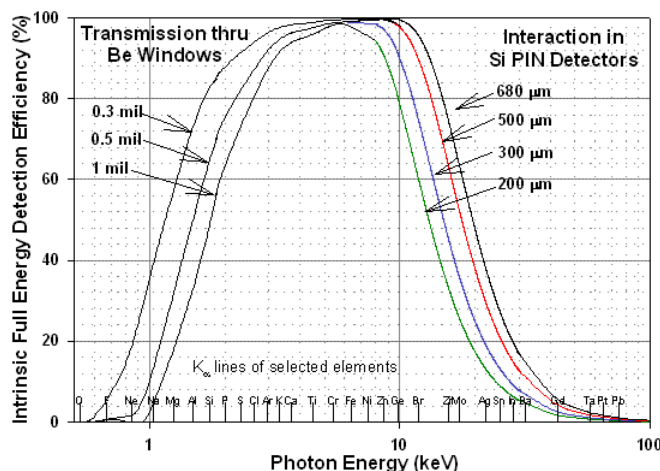


Figure 5: Efficiency curve of the AmpTek XR-100CR

**Caution:** the radioactive source, target and detector are all located inside a lead lined box with a hinged lid. The lead lining provides sufficient shielding so that it is safe to be around the closed box for long periods of time. The source strength is sufficient that it is prudent to minimize the increased exposure when the lid is open. **Open the lid of the box only as necessary to change or remove targets. Be sure to keep the lid closed at all other times.**

### 3.2 The XR-100CR Detector

The XR-100CR semiconductor detector consists of a piece of silicon 300  $\mu\text{m}$  thick. One side of the silicon (anode) is doped with an excess of p-type impurities, and the opposite side (cathode) with an excess of n-type impurities, resulting in the formation of a p-n junction, just as in a conventional

diode. Within the silicon, electrons from the n-type region diffuse across the junction toward the p-type region, and holes from the p-type region diffuse across the junction toward the n-type region. This net migration of charge results in the development of a voltage across the p-n junction, and the formation of a region in which there are no free charge carriers. This region is called the depletion region, and the electric field due to the charge migration reaches a maximum within this region. When electron-hole pairs are created in this region, the electric field acts to sweep the electrons and holes in opposite directions, resulting in a net current through the diode. Since the effect of incident radiation is to create electron-hole pairs in the silicon (one pair for every 3.62 eV of energy in the incident photon), the integral of the current pulse resulting from a photon impinging on the detector will be a measure of that photon's energy. The integral of the current pulse is in turn amplified to produce a shaped output pulse, the (peak) amplitude of which is proportional to the collected charge, and therefore incident photon energy.

The depletion region is clearly the key part of the detector, as electron-hole pairs formed there will move in opposite directions and contribute to the desired energy measurement. Increasing the width of this region increases the probability that the incident radiation will be stopped there and its energy deposited within the region. In order to increase the width of the depletion region, an external bias potential is applied across the detector, with the positive side connected to the cathode, and the negative side connected to the anode. (Note: this bias is applied in the opposite sense of a forward bias, and hence is termed a reverse bias.) The effect of the reverse bias is to pull electrons remaining in the n-type region and holes remaining in the p-type region even further from the p-n junction, resulting in a wider depletion region. In the absence of an external bias, the depletion region is typically some tens of  $\mu\text{m}$  wide, whereas with the bias applied, the depletion region can encompass almost the entire body of the silicon detector. The external bias also acts to increase the strength of the electric field in the depletion region, resulting in increased charge collection efficiency as electrons and holes are less likely to experience recombination or trapping when they are swept out of the region more rapidly.

Electron-hole pairs are also generated by thermal excitations in the silicon. Pairs so generated will add noise to energy measurements and degrade the resolution of the detector. In order to reduce the thermal generation of electron-hole pairs, the detector is cooled to approximately 250 K. The cooling is accomplished with a thermoelectric heat pump. The temperature is monitored by the attached meter, set to measure current. The current is calibrated to readout this kelvin temperature.

A natural question arises at this point: how linear is the XR-100CR detector? It turns out that semiconductor detectors are in general very linear, and the manufacturer of the XR-100CR quotes a linearity of 0.1% over the 1 to 30 keV design range of this device.

To protect the XR-100CR detector it is covered by a beryllium ( $Z = 4$ ) window. To pass lower energy X-rays (several keV), this window must be very thin. The window on our detector is .001" thick. Such a thin window is, of course, very fragile, and being made of Be, also very brittle. **Any contact with the window can easily damage it.** In order to help protect the window, the red plastic cap (with a hole in the end) must be left in place at all times.

### 3.3 Procedure: preliminary setup

Now is a good time to turn on the XR-100CR amplifier/power supply if you have not already done so, along with the rest of the electronics (multimeter and oscilloscope). The power switch for the amplifier/power supply is at the back of the unit on the left side. The RTD (Rise Time

Discriminator) should be on (switch up). Also turn on the DMM. The DMM current reading in amps indicates the detector temperature in kelvins. Typical operating current is  $248 \pm 2 \mu\text{A}$ .

The output pulses from the XR-100CR detector amplifier are positive-going, 0-8 volts peak amplitude. For a given energy incident photon, the amplitude of the output pulse will depend on the amplifier gain setting. The gain is set with the 10-turn potentiometer, and can be varied from 0 to 10.00 units (number in window is the integer, number on dial the fraction). Setting the gain involves a trade-off between the maximum energy you want to detect and the ability to resolve closely spaced lines. The lower the gain, the higher the energy you will be able to detect, but the energy scale will be more compressed and the more difficult it will be to resolve closely spaced lines. At maximum gain, the sensitivity is around 1 volt of output pulse amplitude per keV of incident photon energy. More precisely, with the gain set at 1.00, the detector creates pulses with heights of 1 volt per 8.4 keV of photon energy (approximately).

Before acquiring any spectra, choose an upper bound on the X-ray energy you wish to measure, and set the XR-100CR amplifier gain accordingly. Once this gain is set, it is a good idea to lock the 10-turn potentiometer with the small black lock lever to the right of the dial. As a practical matter, the highest Z target available in the lab that will yield a K spectrum in a reasonable amount of time is gadolinium, with  $K_{\alpha 1}$  energy = 42.98 keV. With this number in mind, you also want to have a reasonable amount of “headroom”, or signal above this maximum value so that you can resolve the highest energy. A good choice is to use 50 keV as a rough maximum energy, and to set the gain so that you get peak heights of about 7.5 volts with this energy. To record a 50 keV photon so that it has a pulse height of about 7.5 volts, a proportional argument gives

$$\frac{\text{Gain} = 1.0}{1 \text{ V}/8.4 \text{ keV}} = \frac{\text{Gain} = x}{7.5 \text{ V}/50 \text{ keV}} \rightarrow x \approx 1.26 \quad (4)$$

Try setting the gain there (or, if it is already at this setting, leave it there) and adjust as necessary. Note that to convert accurately from voltage to energy, you cannot rely on the gain setting. You will need to calibrate the energy scale using the tabulated emission energies of the known samples. Such a calibration should be repeated at the start of each recording session and any time the gain knob is adjusted.

The amplifier output is connected to the input of a digital pulse-processing unit, also made by AmpTek, that performs pulse height analysis. This unit is the AmpTek MCA8000D “Pocket MCA”. *MCA* stands for Multi-Channel Analyzer. The device measures the incoming pulse waveform, determines the heights of the positive-voltage peaks, and creates a pulse-height histogram. The user of the program may select the resolution of the histogram, i.e., the number of bins, along with other parameters. The resulting data can also be analyzed with the unit’s software to find peak locations and widths in terms of the pulse-height voltage, measured in units called “channels.” The range of allowed input voltages is 0–10 volts, and this range may be subdivided in factors of two from 256 up to 8192 channels

Start the AmpTek application **DppMCA** according to the instructions from the TA or lab manager. The default parameters will work well for this experiment. You should consult the Help files accessible from the top menu. In particular, read through the *Overview* and the *Quick Start Guide*.

### 3.4 Acquiring a Spectrum

Before placing any targets in the lead lined box, the TA, Professor or lab manager will go over the hardware arrangement inside the box with you. Again, it is prudent to minimize the time that the

lid is open so as to minimize radiation exposure. After going over the hardware arrangement inside the box, place a target (Fe is a good one to start with) in the holder and close the lid.

Start data collection by clicking the *Stoplight* icon (or select MCA> Start Acquisition from the menu bar).

After a short delay you should see the histogram begin to accumulate. Let the data taking run until you can see clear peaks appear, and then a bit more until the spectrum is well established; aim for at least 100 counts in the peaks you want to analyze. Record the duration of the run. (Note: by default the acquisition continues indefinitely. You may set it up to run for a pre-defined time or pre-defined total number of counts under MCA> Acquisition Setup.)

After you stop the data acquisition (Stoplight again), it is a good idea to save the data set (File>Save As). Then you or your partners may analyze the data by selecting *regions of interest* or *ROIs* dialog box to fit a Gaussian peak shape to the important peaks. Detailed instructions for using the pulse height analysis features are available under Help. Look for *Analyzing Data*.

The data collection and analysis program is free and may be downloaded for use on your own computer. (Note: the software runs on Microsoft Windows only.) To obtain a copy, look for the links on the course website, or perform an internet search on “AmpTek.”

### 3.5 Some advice on data collection and analysis

- Your goal is to measure the characteristic peaks associated with each sample. Start with iron (Fe) to get a strong  $K_{\alpha}$  peak at about 1 volt (assuming the recommended settings). Then collect data for samples by moving away from Fe in terms of  $Z$  both higher and lower.
- For each successive run, pay attention to the new peaks. If you identify the characteristic lines of one sample, you should be able to estimate the location of the peaks you should get with others.
- Think about which peaks in a given spectrum are worth fitting. Don't waste time fitting background peaks (unless you are actually analyzing the background.) The best way to choose is to pay attention to what is new in each spectrum.
- You may load multiple files into the DppMCA software for easy visual comparison. Load a background run before loading one of the sample runs. This makes it easy to spot which peaks are particular to the sample, and also shows likely candidates for the background peak identities.
- The K line may or may not be the strongest peak in a run, depending on the sample. Some samples will show only K lines, some only L lines, and some both.
- As the atomic number gets bigger, the K-lines move up the x-axis. They also separate from each other and get smaller. You can begin to resolve the  $K_{\alpha 1}$  and  $K_{\alpha 2}$  lines. Remember: it is not necessarily the biggest peaks you want, focus on the new peaks that are not in the background even if they are small.
- The very narrow “peaks” at the highest end of the spectrum are false. These peaks simply mark the maximum output of the pulse amplifier. Any pulses which would be higher than this value get clipped off. The MCA records them anyway. *Do not “analyze” these highest “peaks” at about channel 6100.*

- Ignore the “Uncertainty” given in the Peak Information list. This is not related to the peak location or width.
- The overall time of data collection does not matter. What matters is the quality of the statistics for a particular spectrum. If the counting efficiency is low, count longer.

## *References*

- [1] R. Eisberg and R. Resnick, Quantum Physics of Atoms, Molecules, Solids, Nuclei and Particles, 2nd ed., Wiley, New York, 1985, p. 342.
- [2] H. Haken and H. Wolf, The Physics of Atoms and Quanta, Springer, New York, 1996, pp. 314.

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