X-RAY SPECTRA

In this experiment, a number of measurements involving x-rays will be made. The spectrum of x-rays emitted from a molybdenum target will be measured, and the experimental values for the characteristic wavelengths (the $K_{\alpha}$ and $K_{\beta}$ peaks) will be compared with accepted values. The variation of the high frequency (low wavelength) limit of the spectrum with tube voltage will be measured, and a value of $e/hc$ determined. The x-ray absorption spectra of various elements will be measured, and an attempt will be made to verify Moseley’s Law. Finally, x-rays will be used to determine the interatomic spacing of an unknown crystal.

Theory:

X-rays is the name given to that portion of the electromagnetic spectrum with wavelengths in the range $10^{-8}$ to $10^{-11}$ m (quantum energies of 0.1 to 100 keV). X-rays can be produced in tubes such as the one shown schematically in Figure 1.

![An X-ray tube.](image)

Figure 1

Electric current through the filament heats the cathode, and the electrons in the cathode gain enough kinetic energy to overcome their binding to the cathode surface and be released (this is called thermionic emission). The electrons are then accelerated across the evacuated tube by a large electrostatic potential difference $V$, typically several thousand volts, and strike the target, which is the anode. The kinetic energy, $E_K$, acquired by the electrons just before striking the target is given by

$$E_K = eV$$
Upon striking the target the electrons are decelerated and brought essentially to rest in collisions. Each electron loses its kinetic energy, $eV$, because of its impact with the target. Most of this energy appears as thermal energy in the target. (For this reason x-ray targets are usually water-cooled.) In addition, however, there is the production of electromagnetic radiation (x-rays) through the bremsstrahlung process. Bremsstrahlung (German for braking radiation) is the name given to the x-rays produced when a charged particle is accelerated. When an electron strikes the target it will experience deflections of its path due to near collisions with the nuclei of the target atoms. These deflections (accelerations) are accompanied by the emission of one or more bremsstrahlung photons (x-rays). From conservation of energy, the electron loses an amount of kinetic energy in the bremsstrahlung process equal to the energy ($h\nu$) of the photon or photons created. Any electron striking the target may make a number of bremsstrahlung collisions with atoms in the target thereby producing a number of photons. The most energetic photon is produced by an electron whose entire kinetic energy is converted into the electromagnetic energy of a single x-ray photon when the electron is brought to rest in a single collision.

$$E_K = h\nu_{\text{max}}$$

or in terms of wavelength,

$$eV = \frac{hc}{\lambda_{\text{min}}}$$

Thus, the energy distribution of x-ray photons produced in an x-ray tube operated at a given voltage $V$ is continuous, with a well-defined maximum frequency $\nu_{\text{max}}$ or minimum wavelength $\lambda_{\text{min}}$.

Superimposed on the typical continuous x-ray spectrum, with its minimum wavelength dependent on the tube voltage $V$, are sharp spikes dependent on the target material. Figure 2 shows a typical x-ray spectrum.

Figure 2
Collisions between incident electrons and atomic electrons in the target can cause vacancies in the tightly bound inner shells of the target atoms. Electrons from higher (less tightly bound) levels will fill these vacancies, and the energy liberated due to the increased binding energy in the inner shell is emitted as an x-ray photon. Since the binding energies of the different electron shells of an atom are constant for a given type of atom, and depend on the atom, the x-rays emitted are of discrete energy (wavelength) and hence they cause spikes, characteristic of the type of atom, on the continuous x-ray spectrum. These discrete energy x-rays (the x-ray spectrum of the target atoms) due to transitions between the inner electron shells are completely analogous to the optical spectrum observed due to transitions of the outer electrons. Figure 3 shows the energy levels of an atom, and the labelling of the x-rays arising from the various transitions.

The discrete x-rays that will be observed in this experiment are the $K_\alpha$ and $K_\beta$ lines, which arise when a K shell electron which has been ejected due to collision with an incident electron is
replaced by an L shell or M shell electron, respectively. The higher energy M-K transition has a lower probability than the L-K transition, hence the $K\alpha$ and $K\beta$ x-rays have the relative intensity and relative wavelength values shown in Figure 2.

The absorption spectrum of x-rays in a given material has pronounced discontinuities. X-ray absorption occurs mainly due to Compton scattering (Experiment 6) and the photo-electric effect (Experiment 5). In the photo-electric effect a photon is absorbed by an electron. The energy gained from the photon causes the electron to be excited to higher energy levels in the atom, or to be removed from the atom completely. In this experiment, a feature of the absorption spectrum called the K absorption edge is observed for a number of elements by placing them in the x-ray beam. In general, the intensity of x-rays transmitted through an absorber decreases as the energy of the x-rays decreases (i.e. as the wavelength increases). The K edge corresponds to the minimum energy necessary to excite a K electron to the next orbital vacancy in the atom. If the incident x-ray is of any lower energy, it cannot promote a K electron and hence cannot be absorbed by this process. Therefore, there is a discontinuity in the absorption spectrum at this energy (or corresponding wavelength). A typical absorption spectrum is shown in Figure 4.

![Figure 4](image)

If the incident x-ray energy is reduced even further, an L edge corresponding to the minimum energy for excitation of an L electron will occur, and so on.

Determining the K edge energy provides a means of obtaining the energy difference between the K shell and the next shell with vacancies for the atoms of the absorber. For the manganese, iron, and nickel absorbers used in this experiment the M shell ($n = 3$) is the lowest one with vacancies. Therefore the K edge energy should correspond to the transition energy of $n = 1$ to $n = 3$.

According to the Bohr model for single-electron atoms:
\[ |E_1 - E_3| = Z^2 R \left( \frac{1}{17} - \frac{1}{37} \right) \] (5)

Since the absorber atoms are not single-electron atoms, the atomic number, \( Z \), is replaced by \( (Z - b) \) where the number \( b \) accounts for the shielding effects of the electrons.

\[ |E_1 - E_3| = (Z - b)^2 R \left( \frac{8}{9} \right) \] (6)

Since \( |E_1 - E_3| = h \nu \) where \( \nu \) is the frequency of the K edge:

\[ \nu = \frac{8c R (Z - b)^2}{9} \] (7)

where \( b \) is assumed constant for a given transition (i.e. \( K \to M, n = 1 \) to \( n = 3 \)).

Thus, for the \( K \to M \) transition, a plot of \( \sqrt{\nu} \) versus \( Z \) should be linear with a slope of \( \sqrt{\frac{8c R}{9}} \).

A similar relation, between \( K_\alpha \) x-ray frequency and atomic number, was confirmed by Moseley in 1913-14. By measuring the \( K_\alpha \) frequencies for various elements Moseley was able to experimentally determine the atomic number of the element and as a result the correct sequence of the elements in the periodic table was established, and the existence of a number of elements that had not been discovered was predicted.

The other absorber used in this experiment is zirconium. Since the lowest unfilled shell in zirconium is \( n = 4 \) (N shell), its K edge frequency is

\[ \nu = \frac{15c R (Z - b')^2}{16} \]

and so the Zr data point is not expected to lie on the \( \sqrt{\nu} \) vs. \( Z \) line found for the other absorbers.

The x-ray beams in this experiment are analysed according to their wavelength distribution by a crystal spectrometer. The crystal spectrometer is based on the fact that since x-rays are electromagnetic radiation they should exhibit the properties of reflection and interference. The regular arrangement of atoms in a crystal defines families of parallel planes (Bragg planes). X-rays scattered by crystal atoms will undergo constructive interference if: i) their common scattering angle is equal to the angle of incidence of the original beam, and ii) the path length difference between two x-rays scattered from consecutive crystal planes is

\[ 2d \sin \theta = m \lambda \] (8)

where \( m \) is an integer, \( d \) is the crystal plane spacing (0.20134 nm for the LiF crystal used for most of the experiment), \( \theta \) is the incident angle, and \( \lambda \) is the x-ray wavelength. This is known as
Bragg’s equation. In the present experiment the first order, \( m = 1 \), pattern will be observed. Figure 5 shows the constructive scattering of two x-rays incident on a cubic crystal. This scattering is also known as x-ray diffraction.

![Figure 5](image)

**Figure 5**

The schematic design of an x-ray spectrometer based on the wavelength dependence of constructive scattering from a crystal is shown in Figure 6.

![Figure 6](image)

**Figure 6**

The x-ray spectrometer contains two collimating slits which confine the x-rays into a sharp beam. This beam falls upon the single crystal and some of the x-rays are diffracted in accordance with the Bragg equation. The detector mounting is properly geared so that the detector-crystal
angle always equals the angle of the incident x-rays. (i.e. The detector must be moved through an angle $2\theta$ about the rotation axis of the crystal when the crystal is moved through an angle $\theta$.) Note that for a given $m$ value, at each angle $\theta$ there is only one wavelength for which constructive interference occurs. Thus the counting rate recorded by a ratemeter or scaler connected to the detector is a measure of the intensity of the x-rays of the wavelength (and hence energy) selected by the angle of the crystal.

**Apparatus:**

The x-ray unit, manufactured by Philips Electronics, is designed for scientific use and contains a molybdenum target x-ray tube. Figure 7 shows the front controls of the unit.

![Figure 7](image)

Operation of the x-ray unit is fairly straightforward:

**Switching ON:**

1. Open the water tap completely (to cool the x-ray tube).

2. Set slide switch 2 in its lower position.

3. Set the kV and mA (the tube voltage and current) controls in their minimum positions.


The main relay will now be energised and the unit will come on. After the time required for the electronic tubes of the mA stabiliser to warm-up (approximately one minute) the mA meter will show a small increase of the tube current to its normal minimum value.
Required values of tube voltage and current can now be selected.

5. Press pushbutton 2 to open x-ray port to spectrometer. All other ports must remain closed for safe operation.

6. While taking a reading of the spectrometer angle, close the x-ray port by flicking slide switch 2 up and then down.

7. To continue observations press pushbutton 2 again to open the port. When the port is open the orange light below the port will be illuminated.

Switching OFF (at end of experiment):

1. Turn voltage and current controls to minimum (fully counterclockwise).

2. Depress D2 to turn off main power.

3. Close the water tap.

The filters which will be used for the absorption spectra measurements consist of manganese, iron, nickel, and zirconium foils mounted in a wheel in front of the x-ray port. (There is also a vanadium filter which will not be used.) The filter in place is indicated by its atomic symbol being directly above the port. The position of the wheel with no symbol indicates the ‘no filter’ position.

The crystal used in the x-ray spectrometer is lithium fluoride (LiF) which has a crystal plane spacing, \(d\), of 0.20134 nm.

The detector is a Geiger-Muller counter (see the Measurement of Half-life experiment) operated at 500 V which can be connected to either a scaler or ratemeter. Set the scaler high voltage to 500 V. The detector is connected to the terminal labelled INPUT/CAUTION HIGH VOLTAGE of the scaler, and the detector input of the ratemeter.

The operation of the ratemeter is described below. Figure 8 shows the front panel of the ratemeter.

Figure 8
Rotate the Audio-AC Off control clockwise to turn on the unit. Turn the range switch to the HV position and use the bottom scale of the meter to read the value of the high voltage for the detector. The meter should read 500 V. If it does not, rotate the coarse and fine high voltage controls until a voltage of 500 V is attained. The coarse control advances the voltage in approximately 350 volt steps, the fine control fills in between the 350 volt steps.

The Audio-AC Off control can be rotated further clockwise so that counts being registered can be heard on the ratemeter speaker. A count rate scale appropriate to the data being measured can be selected with the Range switch. The centre scale on the meter is used to indicate 0 to 300, 0 to 3000, and 0 to 30,000 counts per minute. The upper scale indicates count rates in the 0 to 1000, 0 to 10,000, and 0 to 100,000 counts per minute ranges.

Set the Time Constant to 1 second.

Set the % Full Scale dial to 100.

NOTE: If the count rate exceeds full scale, the internal alarm light turns on. This may be turned off by pressing the reset button.

Procedure and Experiment:

NOTE: The angles read from the x-ray spectrometer are values of the angular position of the detector with respect to the incident x-ray direction. That is, they are values of $2\theta$ where $\theta$ is the angle of the crystal relative to the incident x-rays. Therefore before wavelengths can be calculated from equation (8) the spectrometer angle readings must be divided by 2.

1. Turn on the x-ray machine following the procedure described in the Apparatus section. Set the x-ray tube high voltage to 40 kV and keep the current at its minimum value. Check that the detector is connected to the ratemeter. Turn on the ratemeter and check that the detector high voltage is set at 500 V (0.5 kV).

2. A multi-sheet Excel workbook named xray.xls is available to aid with the presentation and analysis of the data. Note that you must still do the appropriate sample calculations.

3. Load the workbook xray.xls from the lab manual web page:

4. Once the file has loaded, save it to your home directory (most likely on the h: drive) by selecting Save As... from the File menu.

5. The active worksheet should be ‘Spectra Data’. If this is not the active worksheet, find and select the appropriate tab.

6. The instructions in column A of the Spectra Data worksheet (which are repeated in the following steps 9. through 13.) prompt you for the information that is required to complete the first part of the experiment. Data (pure numbers, no units) are entered in the appropriate
cells of column B. As you collect and enter your data, a formatted table of results and the required graphs are ‘automatically’ produced on other pages of the workbook.

7. Remember to periodically save your work by selecting **Save** from the **File** menu.

8. Using the audio feature of the ratemeter to locate the characteristic peaks, measure the position (spectrometer angle) of the $K_{\alpha}$ peak of the molybdenum (Mo) target to within 0.1° by using the vernier scale. Enter this value in the appropriate cell of the spreadsheet. WITHOUT CHANGING THE POSITION OF THE DETECTOR, disconnect the detector from the ratemeter and connect it to the scaler (high voltage set at 500 V). Measure the x-ray intensity (count rate) for a 0.5 minute (30 sec) count interval. Enter this value in the spreadsheet. (You will have to watch the scaler carefully during this measurement, as the count rate is so high that the display may exceed its upper limit and ‘overflow’.

9. Repeat step 9. for the $K_{\beta}$ peak.

10. With the x-ray port closed, measure the background radiation detected in a 5 minute run. Enter this information in the spreadsheet.

11. You will now measure the x-ray spectrum (distribution of x-ray count rate as a function of spectrometer angle) by making a series of 30 sec (0.5 minute) counts at the spectrometer angles listed in the spreadsheet.

12. Repeat steps 9., 10., and 12. for x-ray tube voltages of 35 kV, 30 kV, and 25 kV.

   *Once you have completed the 25 kV measurements ask the instructor to explain part 2 of the experiment. The procedure for part 2 starts with step 13 on the following pages.*

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**Part 1 Analysis**

Typical graphs are shown on the following pages. Important features of the graphs have been identified.

Do the shapes of your intensity versus spectrometer angle graphs agree qualitatively with Figure 2? Discuss.

Do the positions of the $K_{\alpha}$ and $K_{\beta}$ characteristic peaks appear to depend on x-ray tube voltage? Calculate the experimental $K_{\alpha}$ and $K_{\beta}$ x-ray wavelengths and compare with the accepted values for molybdenum: $K_{\alpha} = 0.0713590$ nm and $K_{\beta} = 0.0632872$ nm.

Determine the minimum wavelength value for each tube voltage. Does the minimum wavelength seem to depend on x-ray tube voltage? Plot $1/\lambda_{\min}$ versus tube voltage. Does this agree with equation (4)? From the slope of the graph determine $e/hc$ and compare with the theoretical value.
40 kV Spectrum

40 kV Cut-off
13. The next part of the experiment involves measuring absorption spectra for various filters in order to determine the K absorption edges and attempt to verify a modified form of Moseley's law (that $\sqrt{\nu_{K\text{edge}}}$ vs. $Z$ should be linear with a slope of $\sqrt{8cR/9}$ for the Mn, Fe, and Ni filters and that the Zr point should lie close to, but not on, the line).

14. Set the x-ray tube voltage to 35 kV. Increase the x-ray tube current to 5 mA. The spectrometer angle ranges (chosen to include the K edges) over which measurements are to be made are given below:

<table>
<thead>
<tr>
<th>Absorber</th>
<th>Z</th>
<th>Spect. Angle (°)</th>
<th>Accepted values for K edge (nm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Mn</td>
<td>25</td>
<td>53 – 59</td>
<td>0.1896</td>
</tr>
<tr>
<td>Fe</td>
<td>26</td>
<td>48 – 54</td>
<td>0.1743</td>
</tr>
<tr>
<td>Ni</td>
<td>28</td>
<td>40 – 46</td>
<td>0.1488</td>
</tr>
<tr>
<td>Zr</td>
<td>40</td>
<td>18 – 22</td>
<td>0.068877</td>
</tr>
</tbody>
</table>

Because the intensity of the x-rays incident on the filters changes with wavelength (angle), the transmitted intensities must be normalised to the incident intensity. This is done by taking an $I_0$ (no absorber) measurement and an $I$ (absorber in) measurement at each angle and calculating $I/I_0$. $I/I_0$ is plotted vs. spectrometer angle ($2\theta$) to determine $2\theta_{K\text{edge}}$. A typical plot would be:
15. Select ‘Moseley Data’ as the active worksheet by clicking on its tab.

16. As before, the instructions in column A of the worksheet prompt you for the information that is required. Data (pure numbers, no units) are entered in the appropriate cells of column B. As you collect and enter your data, a formatted table of results and the required graphs are ‘automatically’ produced on other pages of the workbook.

17. Remember to periodically save your work by selecting Save from the File menu.

\textit{Once you have completed the Zr measurements ask the instructor to explain part 3 of the experiment. The procedure for part 3 starts with step 18 on the following page.}

\textbf{Part 2 Analysis}

From each graph determine the value of the K edge angle. Calculate $\lambda_{\text{Kedge}}$ and $\nu_{\text{Kedge}}$ for each of the filters. Compare your K absorption edge wavelengths with the accepted values (see table on previous page).

Plot $\sqrt{\nu_{\text{Kedge}}}$ vs. Z (atomic number). Is the Zr point collinear with the other three? Explain.

Plot $\sqrt{\nu_{\text{Kedge}}}$ vs. Z for the Mn, Fe, and Ni filters only. Does this agree with the modified Moseley’s law, equation (7)? Compare the slope of the plot to the theoretical value. Determine the value of $b$, the electron shielding correction, from your Mn, Fe, Ni plot.
18. Ask the instructor to replace the LiF crystal with the unknown crystal. The unknown crystal structure is the same as that of LiF, but the interatomic spacing is different. Using the ratemeter, measure the positions of the molybdenum $K_\alpha$ and $K_\beta$ x-rays.

**Part 3 Analysis**

Using equation (8) and the $K_\alpha$ and $K_\beta$ wavelengths determined previously, calculate an average value for the interatomic spacing of the unknown crystal.