X-ray Spectroscopy

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1 Abstract

In this experiment the X-Ray Spectrum was investigated, as was X-Ray Attenuation. X-ray spectrums for a range of accelerating voltages U were obtained. It was shown that $U\lambda_{min}$ is constant, where λ_{min} is the minimum wavelength produced by the decelerating electrons. A value for Planck's Constant of $5.5 \pm 0.1 \times 10^{-34}$m^{2}kg s$-1$ was calculated, which is of the correct order of magnitude of the accepted value of 6.626×10^{-34}m^{2}kg s-1. The photon wavelengths and energies for the K_α and K_β peaks were measured to be 65±2pm and 57±2pm, and 19.2±0.6keV and 21.8 ± 0.7keV respectively. These values are the same order of magnitude and compare well to the theoretical values of 17.1keV and 20.3keV, and the accepted values of 71pm and 63pm, or 17.5keV and 19.6keV respectively.

The K-Edge Absorption Energies E_K of Zirconium, Molybdenum, Silver and Indium were found to be 19.7±0.3keV 22.1±0.4keV 29.3±0.7keV and 32.3±0.8keV corresponding to wavelenghts of 63±1pm 56±1pm 4±1pm2 and 38±1pm for respectively. It was shown that $E_K \propto (Z - \sigma_K)^2$ where Z is the Atomic Number and σ_K is the K Shell Screening Parameter which was found to be 8.0 ± 0.8, and a value for the Rydberg Constant R of $(15.6 \pm 0.2) \times 10^6$m^{-1} was measured. This is of the right order of magnitude as the accpeted value is 10.97×10^6m^{-1}.

The Mass Attenuation Coefficients of Aluminium, Iron, Copper and Zirconium were found to be 0.29±0.03m^2 kg^{-1}, 0.93±0.01m^2 kg^{-1}, 1.01±0.07m^2 kg^{-1} and 2.67±0.12m^2 kg^{-1} respectively, which are comparable to the accepted values of 0.11m^2 kg^{-1}, 0.77m^2 kg^{-1}, 1.08m^2 kg^{-1} and 2.51m^2 kg^{-1}. Finally the dependence of the Absorption Cross-Section σ_a on the Atomic Number Z was found to be $\sigma_a \propto Z^4$ for X-rays in the range of 10keV to 40keV.

2 Introduction & Theory

2.1 The X-ray Spectrum

An *X-ray Spectrum* consists of sharp lines called the *Characteristic Spectrum* over a *Continuous Spectrum* extending to a minimum wavelength λ_{min}.

This continuous spectrum is due to radiation produced by the decelerating incident electrons. The minimum wavelength is thus given by

$$\lambda_{min} = \frac{hc}{e}U \tag{1}$$

where $h \simeq 6.626 \times 10^{-34}$m^{2}kg s^{-1} is Planck's Constant, $c \simeq 2.9979 \times 10^8$ms^{-1} is the speed of light, and U is the tube voltage. This is because the electrons must have a minimum energy $E = eU$ and the photons produced have energy

$$E = \frac{hc}{\lambda} \tag{2}$$

The characteristic spectrum however is due to electron transitions. Incident electrons knock off inner shell electrons from the target atoms, and outer shell electrons then fall to fill the vacancy with the emission of a photon. This gives rise to two peaks, K_α and K_β, due to electron tranissitions from the L to the K shell, and from the M to the K shell respectively. The binding energy of the electrons may be found using the Modified Bohr Model of the atom, giving

$$E_n = \frac{-Rhc}{n^2} Z_{eff}^2 \tag{3}$$

where

$$Z_{eff} = Z - \sigma_m$$

where $R \simeq 10.97 \times 10^6 \text{m}^{-1}$ is the Rydberg Constant, h and c are as above, n is the shell number, Z is the atomic number, and σ_m is the screening constant. Thus we get

$$E_{K_\alpha} = \frac{3Rhc}{4} Z_{eff}^2$$

and

$$E_{K_\beta} = \frac{8Rhc}{9} Z_{eff}^2$$

For an electron falling from the L shell to the K shell we can set

$$\sigma_m = 1$$

. This is because the K shell only has two electrons, so if one is knocked off due to an incident electron, there is only one electron left to screen the nucleus.

For X-rays diffracting off a crystal the wavelength λ of the incident rays is related to the angle of reflection β by the *Bragg Law*

$$n\lambda = 2d \sin(\beta) \tag{4}$$

where n is the order of the image and d is the atomic spacing. For an NaCl crystal $2d = 563 \times 10^{-12}$m and $n = 1$.

2.2 X-Ray Absorption and Scattering

Radiation incident on a slab of material will have reduced intensity due to *Absorption* and *Scattering* according to

$$T = \frac{I}{I_0} = \frac{R}{R_0} \tag{5}$$

where T is the *Transmittance* and I,I_0,R and R_0 are the resultant and initial intensities and count rates respectively.

For a slab of thickness x with n atoms per unit volume, if photons within an area σ, known as the *Removal Cross Section*, are removed from the incident radiation due to either absorption or scattering, then the radiation intensity is reduced according to

$$I = I_0 \exp^{-\sigma n x}$$

or

$$I = I_0 \exp^{-\mu x} \tag{6}$$

where $\mu = \sigma n$ is the *Linear Attenuation Coefficient.*

Thus from equations (5) and (6)

$$\mu = \frac{-\ln(T)}{x} \tag{7}$$

Now,

$$n = \frac{N_A \rho}{A}$$

where N_A is Avagadro's Number, and ρ is the density of the material expressed in grams, giving

$$\sigma = \frac{A}{N_A} \frac{\mu}{\rho} \tag{8}$$

where μ/ρ is the *Mass Attenuation Coefficient.*

Photons can be removed by both absorption and scattering, giving

$$\sigma = \sigma_a + \sigma_s \tag{9}$$

where σ_a and σ_s are the partial absorption and scattering cross sections respectively. For electrons in the range of 10 to 40 keV scattering does not contribute much and so

$$\sigma_s = \frac{0.02A}{N_A} \tag{10}$$

For electrons in the range of 10 to 40 keV absorption causes the *Photoelectric Effect.* Thus incident photons must have energies $E = h\nu$ greater than or equal to the binding energy of a K shell electron. Hence, when the photon energy reaches this value we get a jump in the absorption called the *K-Absorption Edge*, where

$$E_K = \frac{hc}{\lambda_K} = Rhc(Z - \sigma_K)^2 \tag{11}$$

or

$$\lambda_K^{-1/2} = \sqrt{R}Z - \sqrt{R}\sigma_K \tag{12}$$

There is also have a rapid increase in absorption with the atomic number, and for energies in the given range

$$\sigma_a \propto Z^p$$

or

$$\sigma_a = cZ^p$$

where c is some constant. Therefore

$$\ln(\sigma_a) = p\ln(Z) + ln(c) \qquad (13)$$

3 Experimental Method

3.1 The X-Ray Spectrum

The equipment was set to Coupled mode to alow for a $\theta - 2\theta$ scan as per the *Bragg Geometry*.

The tube voltage U, tube current I, measurement time interval Δt and step angle $\Delta\beta$ were set to 35kV, 1mA, 5s and 0.1^o respectively.

The limits of the angle β were set to 3^o and 10^o. A lower limit of zero was not used as this would not allow for any diffraction.

The software was run, and the NaCl crystal was adjusted in orientation for a few trial diffractions to give the best peaks.

Scans were taken for a range of values of U, with each successive scan superimposed on the last.

The minimum wavelengths λ_{min} and the K_α and K_β wavelengths were found using equation (4) using the measured values of β.

The corresponding energies E_{max} and E_{K_α} and E_{K_β} were then calculated using equation (2).

The values of $U\lambda_{min}$ were calculated and compared, and Planck's Constant was found.

The theoretical values for E_{K_α} and E_{K_β} were calculate using equation (3) on setting $\sigma_m = 1$.

3.2 The K-Absorption Edge

The lower and upper limits of the angle β were set to 2^o and 12^o respectively.

Using $U = 35$kV, and the same values for I, Δt and $\Delta\beta$ the X-ray spectrum was recorded.

This was then repeated with Zr, Mo, Ag and In foils covering the beam, superimposing all spectra on the original.

The β scale was converted to a λ scale and the data was re-plotted.

The data was then converted to give a graph of the tranmission T versus the wavelength λ.

The K-Edge wavelengths λ_K were found and the energies E_K were then calculated using equation (2).

A graph was plotted to verify equation (11). From this values for R and σ_K were found using equation (12).

3.3 The Mass Attenuation Coefficient and Absorption Cross-Section

A suitable choice of angle β was chosen to give a photon whose wavelength was away from an absorption edge using equation (4).

$\Delta\beta$ was set to zero and Δt was set to 20s, and using the same values for U and I as above, and this choice of β, the count rate without foil R_0 was measured.

This was then repeated with Al, Fe, Cu and Zr foil covering the beam.

Equation (7) was used to find μ, and the Mass Attenuation Coefficient μ/ρ was then calculated for each foil.

The Absorption Cross Section σ_a was then found for each foil using equations (8), (9) and (10).

Finally, a graph of plotted to verify equation (13) and the value of p was found.

4 Results & Analysis

4.1 The X-Ray Spectrum

The following spectrum for a Molybdenum X-ray source was obtained for tube potential values of 35, 30, 25 and 20 kV respectively

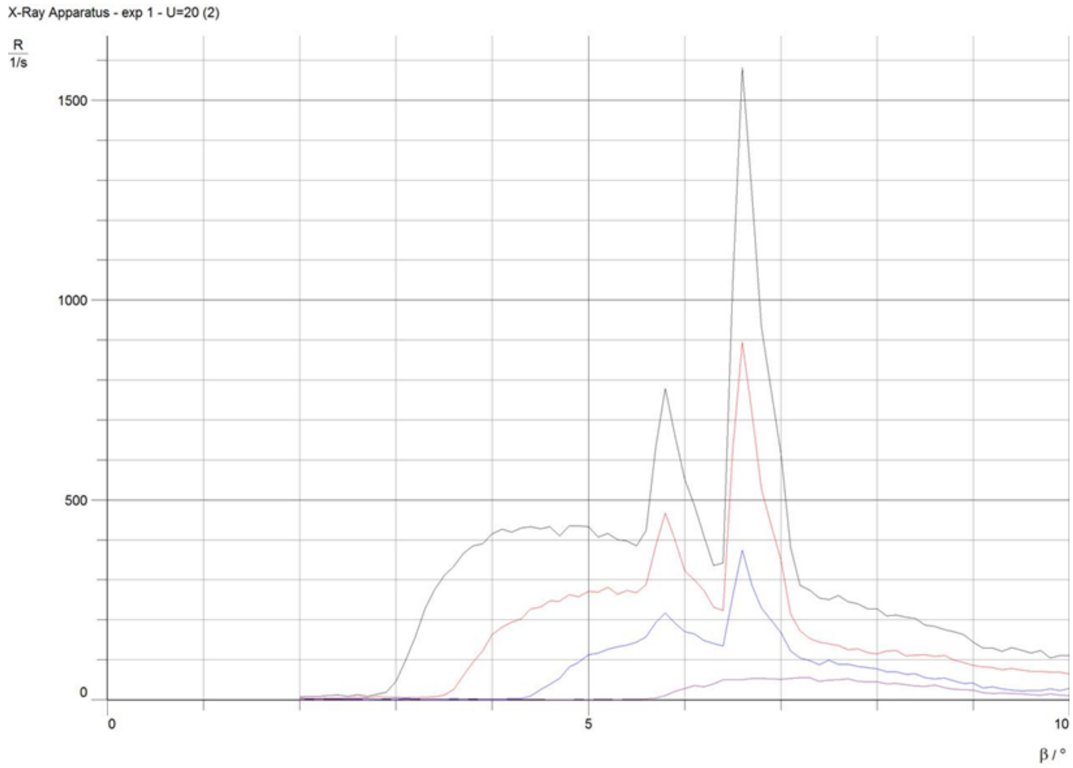

Figure 1: X−Ray Spectrum

From this the following data was calculated

Tube Voltage, U (kV)	35	30	25	20
Minimum Wavelength, λ_{min} (pm)	28±2	34±2	42±2	52±2
K_α Wavelength, λ_{K_α} (pm)	65±2	65±2	65±2	N/A
K_β Wavelength, λ_{K_β} (pm)	57±2	57±2	57±2	N/A
Maximum Energy, E_{max} (keV)	44±3	36±2	29±1	24±1
K_α Energy, E_{K_α} (keV)	19.2±0.6	19.2±0.6	19.2±0.6	N/A
K_β Energy, E_{K_β} (keV)	21.8 ± 0.7	21.8 ± 0.7	21.8 ± 0.7	N/A
$U\lambda_{min}$ (V m $\times 10^{-6}$)	1.00±0.07	1.03±0.06	1.06±0.05	1.04±0.04

These give values for Planck's Constant of $5.3 \pm 0.4 \times 10^{-34}\text{m}^2\text{kg}\,\text{s}{-1}$, $5.5 \pm 0.3 \times 10^{-34}\text{m}^2\text{kg}\,\text{s}{-1}$, $5.6 \pm 0.3 \times 10^{-34}\text{m}^2\text{kg}\,\text{s}{-1}$ and $5.6 \pm 0.2 \times 10^{-34}\text{m}^2\text{kg}\,\text{s}{-1}$, with an average value of $5.5 \pm 0.1 \times 10^{-34}\text{m}^2\text{kg}\,\text{s}{-1}$.

The theoretical values for E_{K_α} and E_{K_β} were found to be 17.1keV and 20.3keV respectively.

4.2 The K-Absorption Edge

The following graph was obtained for a range of foils covering the X-ray beam

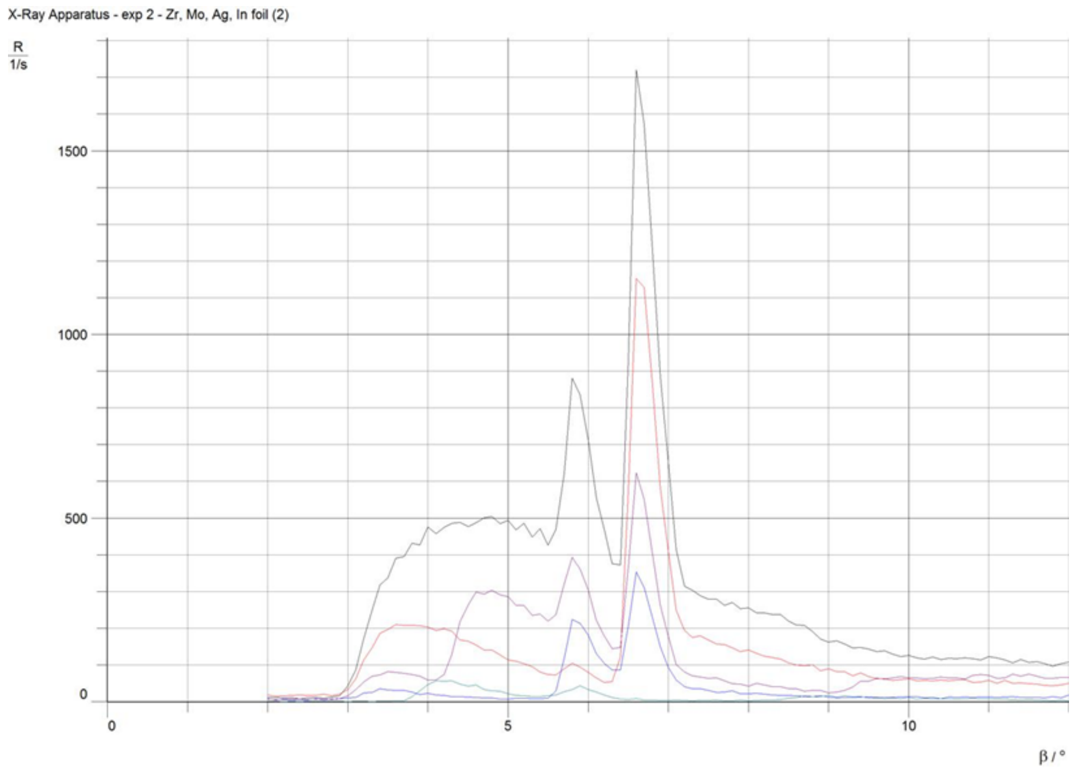

Figure 2: X−Ray Spectrum using Zr, Mo, Ag and In foil

On changing to a wavelength scale the following graph was obtained

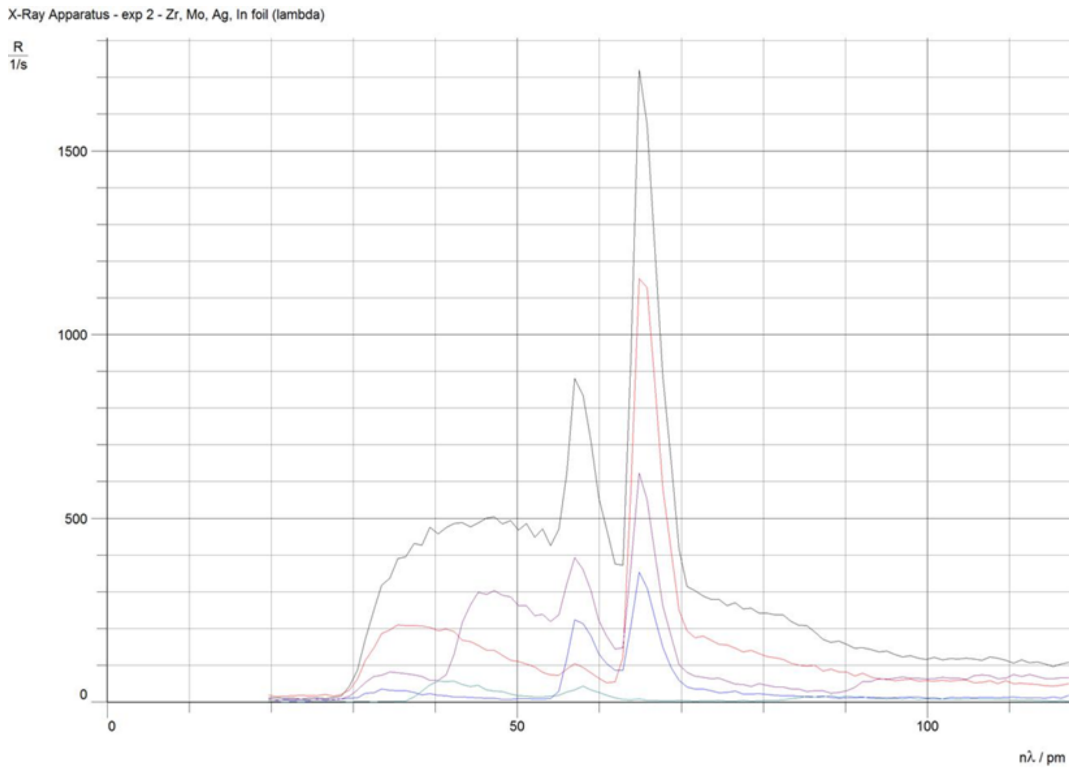

Figure 3: Wavelength Plot of the X−Ray Spectrum using Zr, Mo, Ag and In foil

The following values of the transmittance T for a range of values of incident angle β were calculated

Wavelength λ (pm)		Zirconium Zr	Molybdenum Mo	Silver Ag	Indium In
33.4	Calculated Values	0.5874	0.1093	0.2299	0.0041
	Plotted Values	0.587	10.9	0.230	0.004
43.3	Calculated Values	0.3455	0.0271	0.0637	0.0988
	Plotted Values	0.346	0.027	0.445	0.099
54.1	Calculated Values	0.1736	0.0216	0.5143	0.0360
	Plotted Values	0.174	0.022	0.514	0.036
73.6	Calculated Values	0.6006	0.1108	0.2270	0.0125
	Plotted Values	0.601	0.111	0.227	0.013
84.3	Calculated Values	0.4758	0.0781	0.1667	0.0561
	Plotted Values	0.476	0.078	0.167	0.056

Finally, a graph of the tranmission was obtained

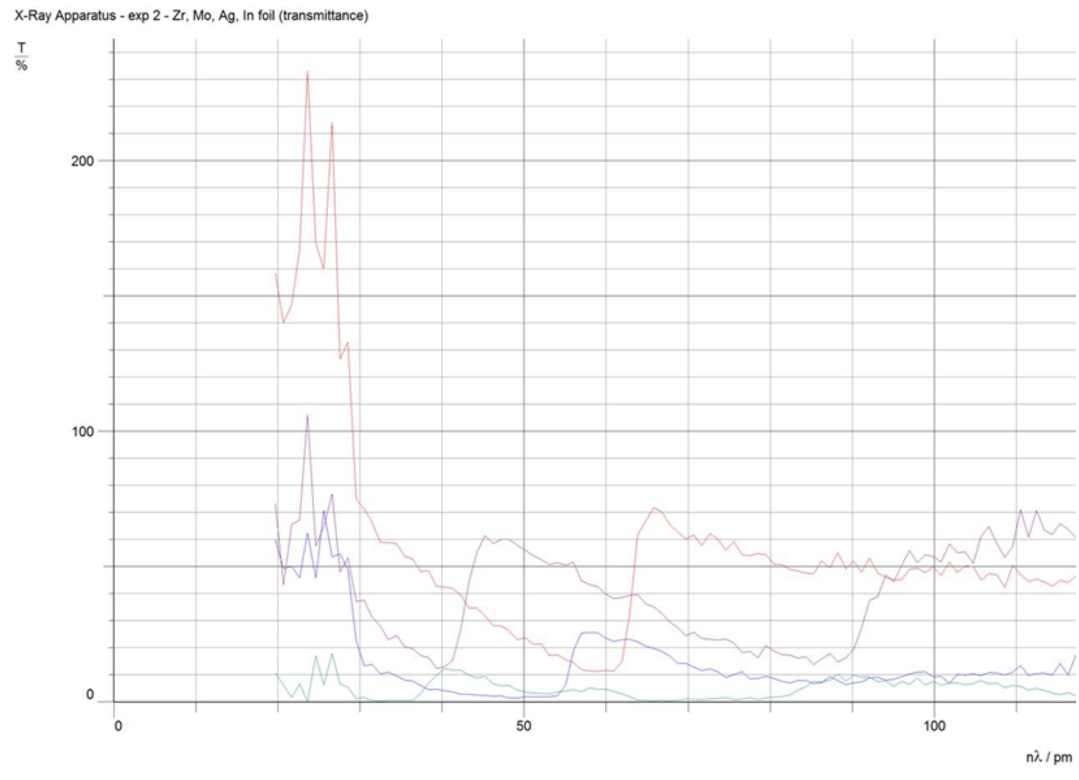

Figure 4: Transmittance Plot of the X−Ray Spectrum using Zr, Mo, Ag and In foil

The following values of λ_K and E_K were calculated for each foil

	Zirconium Zr	Molybdenum Mo	Silver Ag	Indium In
K-Edge Wavelength, λ_K (pm$\pm$1)	63	56	42	38
K-Edge Energy, E_K (keV)	19.7$\pm$0.3	22.1$\pm$0.4	29.3$\pm$0.7	32.3$\pm$0.8

The following graph was then plotted

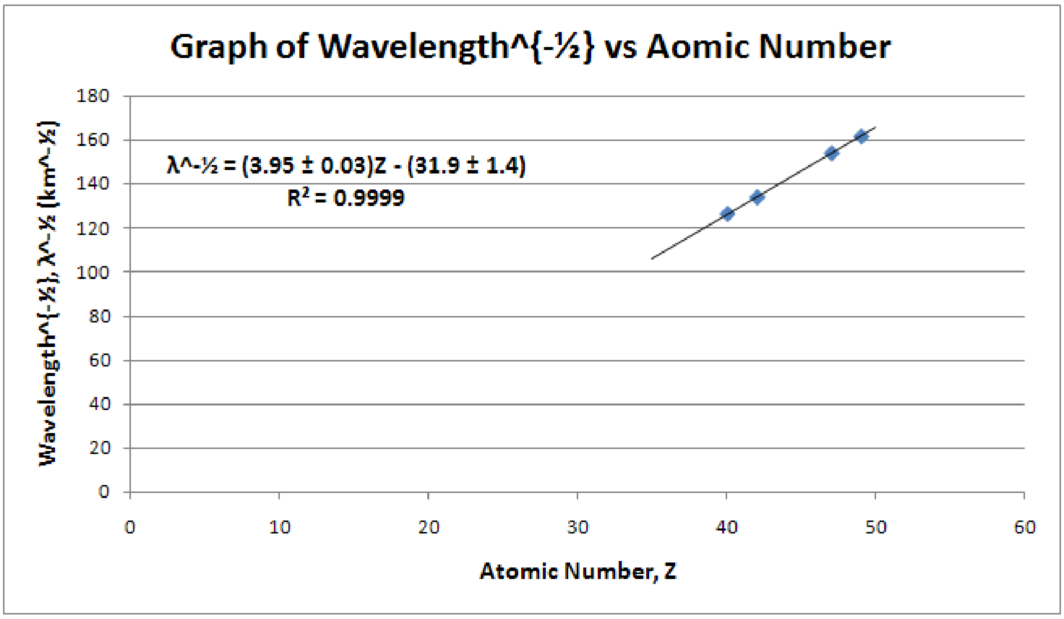

Figure 5: Graph of Wavelength versus Atomic Number

4.3 The K-Absorption Edge

The choice of $\lambda = 41.3$pm giving $\beta = 4.2^o$ was made.

The initial count rate was found to be 34.9±0.9.

The count rates for the Al, Fe, Cu and Zr foils were 23.7±0.8 0.89±0.04 17.6±0.7 and 34.2±0.9*respectively*.

The following values of μ/ρ and σ_a were then calculated

	Aluminium Al	Iron Fe	Copper Cu	Zirconium Zr
Mass Attenuation Coefficient, μ/ρ (m^2 kg^{-1})	0.29±0.03	0.93±0.01	1.01±0.07	2.67±0.12
Absorption Cross-Section, σ_a ($m^2 \times 10^{-27}$)	4±1	68±1	94±8	375±18

The following graph was then plotted

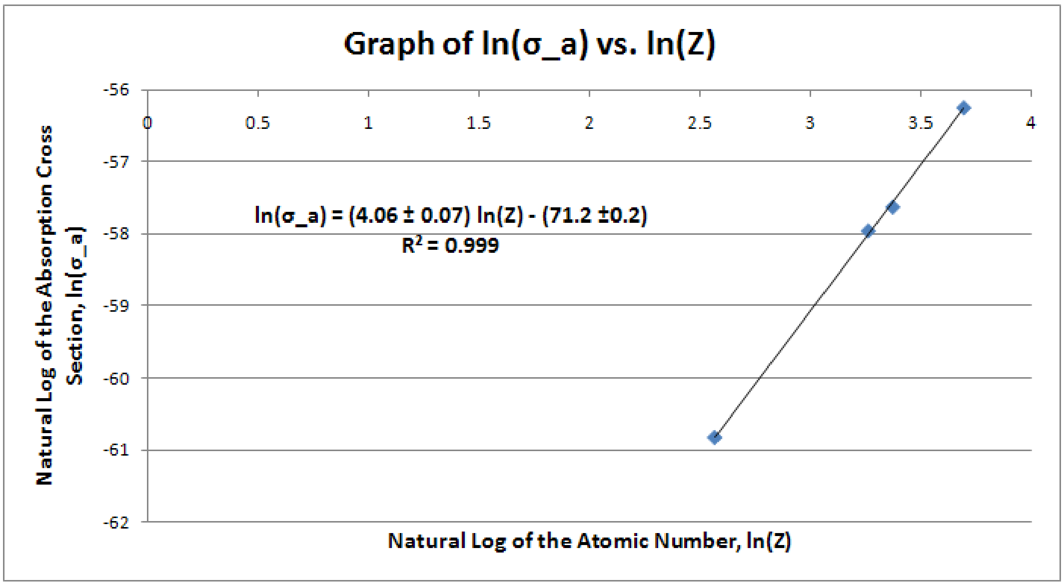

Figure 6: Graph of the Natural Log of the Absorption Cross-Section versus the Natural Log of the Atomic Number

5 Error Analysis

The standard error in the diffraction angles was taken to be $\pm 0.1^o$. The error in the minimum wavelenghts λ_{min} and the K_α and K_β wavelenghts were then

$$\Delta\lambda_i = \lambda_i \times (\sin(\beta + \Delta\beta) - \sin(\beta - \Delta\beta)$$

Thus the error in the corresponding energies E_{max} and E_{K_α} and E_{K_β} were

$$\Delta E_i = E_i \times \sqrt{\left(\frac{\Delta\lambda_i}{\lambda_i}\right)^2}$$

Likewise, the errors in $U\lambda_m in$ and h were given by

$$\Delta(U\lambda_m in) = U\lambda_m in \times \sqrt{\left(\frac{\Delta\lambda_m in}{\lambda_m in}\right)^2}$$

$$\Delta h = h \times \sqrt{\left(\frac{\Delta(U\lambda_m in)}{U\lambda_m in}\right)^2}$$

The error in the average value for h was taken as the standard deviation of the range of values. The standard error in the absorption edge wavelengths λ_K were taken to be ± 1pm. The error in the absorption edge energies E_K was then

$$\Delta E_K = E_K \times \sqrt{\left(\frac{\Delta\lambda_K}{\lambda_K}\right)^2}$$

The error in the Rydberg constant R was given by

$$\Delta R = R \times \sqrt{2} \times \sqrt{\left(\frac{\Delta m}{m}\right)^2}$$

where m is the slope of the corresponding graph, while that for the screening parameter σ_K was given by

$$\Delta\sigma_K = \sigma_K \times \sqrt{\left(\frac{\Delta m}{m}\right)^2 + \left(\frac{\Delta c}{c}\right)^2}$$

where c is the constant of the corresponding graph. The error in the count rates were

$$\Delta R = \frac{\Delta C}{t} = \frac{\sqrt{C}}{t}$$

where $\Delta C = \sqrt{C}$ is the error in the court and t is the count time. This error can be reduced by taking longer count time. For the error in the mass attenuation coefficients the following relations were used

$$\Delta T = T \times \sqrt{\left(\frac{\Delta R_0}{R_0}\right)^2 + \left(\frac{\Delta R}{R}\right)^2}$$

$$\Delta\mu = \frac{1}{x}\frac{1}{T}\Delta T$$

$$\Delta\frac{\mu}{\rho} = \frac{\mu}{\rho} \times \sqrt{\left(\frac{\Delta\mu}{\mu}\right)^2}$$

The error in the absorption cross-sections were then given by

$$\Delta\sigma_a = \sigma_a \times \sqrt{\left(\frac{\Delta\mu}{\mu}\right)^2}$$

6 Conclusions

6.1 The X-Ray Spectrum

The K_α and K_β Peak Wavelengths were found to be 65±2pm and 57±2pm respectively, corresponding to energies of 19.2±0.6keV and 21.8 ± 0.7keV. These is of the right order of magnitude and compares well to the expected values of 71pm and 63pm, or 17.5keV and 19.6keV resepectively

There were no values for λ_{K_α}, λ_{K_β}, E_{K_α} or E_{K_β} for a tube potential of 20kV as there were no well defined peaks in the spectrum.

The values of $U\lambda_{min}$ were found to be within experimental error of each other, as were the values for Planck's Constant h. The average value wa found to be $5.5 \pm 0.1 \times 10^{-34}$m^{2}kg s$-1$. This is of the right order of magnitude of the accepted value of 6.626×10^{-34}m^{2}kg s-1.

The theoretical values for the K_α and K_β peak energies of 17.1keV and 20.3keV respectively compare well to the measured values of 19.2±0.6keV and 21.8 ± 0.7keV, as they are of the same order of magnitude. The discrepancy is due to the choice of $\sigma_m = 1$ in both cases, as in reality the screening parameter is different for L to K and M to K transitions.

6.2 The K-Absorption Edge

It was seen that the values of Transmissions T plotted matched perfectly those analytic values.

The K-edge Absorption Energies were found to be 19.7±0.3keV 22.1±0.4keV 29.3±0.7keV and 32.3±0.8keV corresponding to wavelengths of 63±1pm 56±1pm 4±1pm2 and 38±1pm for Zirconium, Molybdenum, Silver and Indium respectively.

From the graph plotted equation (11) is verified. Furthermore, the Rydberg constant R was calculated to be $(15.6 \pm 0.2) \times 10^6 \mathrm{m}^{-1}$ while the K shell screening parameter σ_K was found to be 8.0 ± 0.8. This value for R is of the same correct order of magnitude as the accepted value of $10.97 \times 10^6 \mathrm{m}^{-1}$.

6.3 The Mass Attenuation Coefficient and Absorption Cross-Section

The Mass Attenuation Coefficients of Aluminium, Iron, Copper and Zirconium were found to be $0.29 \pm 0.03 \mathrm{m}^2\ \mathrm{kg}^{-1}$, $0.93 \pm 0.01 \mathrm{m}^2\ \mathrm{kg}^{-1}$, $1.01 \pm 0.07 \mathrm{m}^2\ \mathrm{kg}^{-1}$ and $2.67 \pm 0.12 \mathrm{m}^2\ \mathrm{kg}^{-1}$ respectively. These are comparable to the accepted values of $0.11 \mathrm{m}^2\ \mathrm{kg}^{-1}$, $0.77 \mathrm{m}^2\ \mathrm{kg}^{-1}$, $1.08 \mathrm{m}^2\ \mathrm{kg}^{-1}$ and $2.51 \mathrm{m}^2\ \mathrm{kg}^{-1}$ respectively[1].

The Absorption Cross-Sections were then found to be $\pm 1 \mathrm{m}^2 \times 10^{-27}$, $68 \pm 1 \mathrm{m}^2 \times 10^{-27}$, $94 \pm 8 \mathrm{m}^2 \times 10^{-27}$ and $375 \pm 18 \mathrm{m}^2 \times 10^{-27}$ respectively.

Finally, the graph obtained shows that indeed $\sigma_a \propto Z^p$ where $p = 4.06 \pm 0.07$ as expected for the energy range in use.

[1]Compilation of X-Ray Cross Sections, Lawrence Livermore National Laboratory Report UCRL-50171 Section II Revision I (1969)