X-Ray Spectroscopy Lab Ed Heeney

Edward Heeney, lab partner: Megan Haughton-Martinez

February 2024

Contents

1 Abstract

The following Lab has to experiments within. In the first experiment the x-ray spectra for a molybdenum anode was successfully recorded at accelerating voltages of 20-35 keV. $\lambda_{min} = const$ was found to be $11.2\mu meV \pm 0.0025\mu meV$. This allowed for a calculated Planck's constant of $6.39 \cdot 10^{-34}/s \pm 8.33 \cdot 10^{-34}/s$, which had a difference of $0.29 \cdot 10^{-34} \pm 8.33 \cdot 10^{-34}/s$. The first experiment also included acquiring measurements of the $K_{\alpha,\beta}$ lines in the characteristic spectrum of molybdenum. These were found to be 17.58±0.013 keV and 19.75±0.009 keV respectively, which varied by 0.38±0.013 keV and 0.65±0.009 keV from the calculated values in the theory section respectively.
The second experiment had itself two sections. The first of which included determining the energy at the K absorption edge for zirconium, molybdenum and silver, as well as the photon energies of the alpha and beta K lines i the characteristic spectrum. (Indium was not included as a software failure deleted the date required for this). The photon energies of the alpha and beta K lines in the characteristic spectrum of molybdenum were found to be 17.58±0.013 keV and 19.75±0.009 keV respectively, which varied by 0.38±0.013 keV and 0.65±0.009 keV from the calculated values in the theory section respectively.The energy at the K absorption edge for zirconium, molybdenum and silver were found to be 18.06 keV±0.11keV, 20.37 keV±0.13keV and 25.74 keV±0.15keV respectively. The Rydberg constant R was calculated to be $R \approx 1.14 \cdot 10^7/m \pm 1.03 \cdot 10^7/m$. In the second part of experiment two first the mass attenuation of foils were investigated, as it relates to the absorption cross section (In writing up it was realised some foils were used in place of the foils mentioned in the lab-book, however similar results were still able to be obtained).The mass attenuation coefficients of the foils were calculated to be $2.38m^2/kg \pm 0.001m^2/kg$ for Zr, $2.73m^2/hg \pm 0.0004m^2/kg$ for Mo, $40.5m^2/kg \pm 0.002m^2/kg$ for Ag, and $2.36m^2/kg \pm 0.017m^2/kg$ for indium. The absorption cross section for Zirconium, Molybdenum, Silver and indium was calculated. it was found that $\sigma_a = 216.55(b/N_a) \pm 0.13(b/N_a)$ for zirconium, $\sigma_a = 261.44(b/N_a) \pm 0.03(b/N_a)$ for Molybdenum, $\sigma_a = 351.87(b/N_a) \pm 0.25(b/N_a)$ for Silver, and $\sigma_a = 270.46(b/N_a) \pm 1.98(b/N_a)$ for Indium. the relationship between σ_a and its Z number was investigated and it was found that $\sigma_a \propto Z^{2.84 \pm 0.47}$, which differed from the expected $\sigma_a \propto Z^4$.

2 Theory

2.1 X-ray Spectra

As seen in Figure 1, x-ray spectrum consists of two spectra; the characteristic spectrum, which are the peaks, and the continuous spectrum, which is the smooth curve, which carries on until λ_{min}.

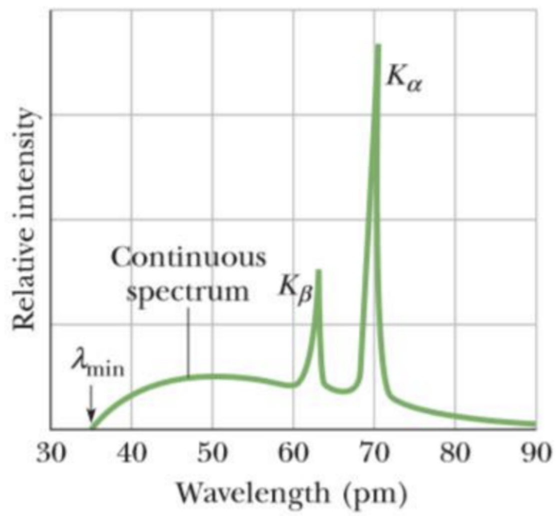

Figure 1: X-Ray Spectrum

The *continuous spectrum* is caused by the declaration of electrons when hitting the target. Because of energy conservation, the kinetic energy is converted into heat and x-rays. When the electron comes into contact with a target atom, it gets close to the nucleus. As the electron is negatively charged, and the nucleus is positive, the nucleus will release an electrostatic force. This force reduces the speed of the electron, and diminished its energy. This causes the x-rays to not have one specific frequency, but a spectrum. [2].The minimum wavelength λ_{min} can be shown as follows

$$E = eV = hf = \frac{hc}{\lambda_{min}} \implies \lambda_{min} = \frac{hc}{eV} = const. \tag{1}$$

The *characteristic spectrum* is caused by the transition of electrons between inner and outer shells of an atom. Outer shell electrons fill a vacancy of the inner shell. This process causes an x-ray to be released, which is characteristic to each element.[3]. THE vacancy of an inner electron is opened up by in incident electron knocking an electron out on an inner shell, say K, of a target atom. If we imagine two electrons from an inner shell K have been displaced so an electron from shells L and M filling the vacancy. These electron transitions can be seen in Figure 2.

The photon energies $E_{K\alpha}$, $E_{k\beta}$ correspond to the photon energies from the $L \to K$ and $M \to K$ transmissions, respectively. We can estimate these energies using a modified version

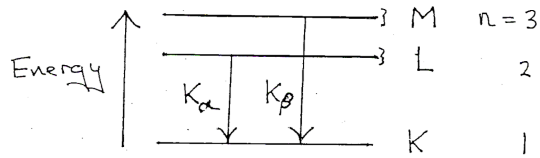

Figure 2: Electron transmission between K and L and k and M levels

of Bohr's Law. The binding energies are then estimated as:

$$E_n = -\frac{RhcZ_{eff}^2}{n^2} \tag{2}$$

n represents the quantum number and

$$Z_eff = Z - \sigma_m \tag{3}$$

σ_m accounts for the partial screening of the nuclear charge and R is the Rydberg constant. From this we can see that

$$E_{k\alpha} = E_2 - E_1 = -(\frac{Rhc(42-1)^2}{2^2}) + (\frac{Rhc(42-1)^2}{1^2}) \approx 17.2keV \tag{4}$$

and

$$E_{k\beta} = E_3 - E_1 = -(\frac{Rhc(42-1)^2}{3^2}) + (\frac{Rhc(42-1)^2}{1^2}) \approx 20.3keV \tag{5}$$

We can compare these results to the results obtained by the Lawrence Berkeley Nation Observatory in The United States.[1]

42 Mo	17,479.34	17,374.3	19,608.3

Figure 3: Molybdenum $k_{E\alpha}$ and $k_{E\beta}$ in eV

The first 2 columns are $k_{E\alpha}$ and the third column is $k_{E\beta}$

Finally for a large crystal structure Bragg's law gives the angles for coherent scattering of waves from it.

$$2dsin(\beta) = n\lambda \tag{6}$$

where n is the order number, d is the inter planar spacing, λ is the wavelength and β is the angle of incidence

2.2 X-Ray Absorption and Scattering

For a thin homogeneous slab, with n atoms per unit volume, with radiation of intensity I incident on it can be shown as in Figure 4

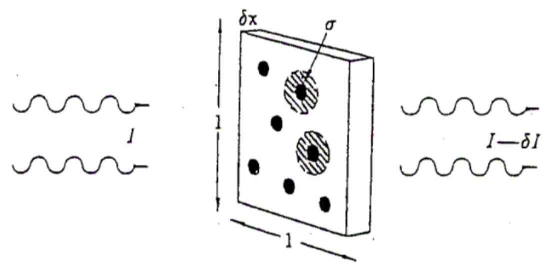

Figure 4: Incident ray upon thin homogeneous slab

From Figure 4 we can see that

$$-(\frac{\delta I}{I} = \sigma \; n \; \delta \; x) \tag{7}$$

where σ is the removal cross section. Then by integrating over slab thickness x, and calling the incident intensity I_0

$$I = I_0 e^{-\sigma \; n \; x} \tag{8}$$

and by letting $\mu = \sigma \; n$, which is the linear attenuation coefficient

$$I = I_0 e^{-\mu \; x} \tag{9}$$

$n = \frac{N_a \rho}{A}$, where N_a is Avagrado's Number and A is the atomic weight of a material with density ρ. Using this we can equate σ as follows:

$$\sigma = \frac{\mu}{n} = \frac{\mu A}{\rho N_a} \tag{10}$$

With A and N_a being constants and as σ is independent of the density of the medium so is $\frac{\mu}{\rho}$.
$\sigma = \sigma_a + \sigma_s$ as photons can be removed from beam by *absorption* or *scattering*, so σ_a and $\sigma_s$are the associated partial cross sections, respectively. Absorption of energy can take place through four methods; the photoelectric effect, Compton scattering, Pair production, ad Rayleigh Scattering. The photoelectric effect dominates energies between binding energies, with Rayleigh scattering well below, and Compton scattering well above. Pair production happens only at 1 MeV. The range we will be working is is about 10-40 keV, so we will focus on the photoelectric effect.
For the ejection of a K shell electron

$$h\nu \geq \text{binding energy of K shell electron} \tag{11}$$

This implies a jump in photoelectric effect cross section and attenuation after the photon has reached this binding energy. We can see this in

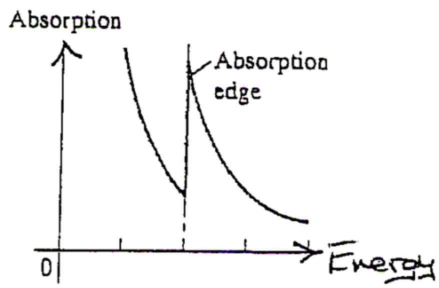

Figure 5: Absorption edges in a Solid

The discontinuity of the K shell is called the K-edge. At this edge the energy is as follows;

$$E_k = \frac{hc}{\lambda_k} = Rhc(Z - \sigma_k) \tag{12}$$

This is what Moseley's empirical relation was based on

$$\sqrt{\frac{1}{\lambda_k}} \propto Z - \sigma_k \tag{13}$$

Also seen in Figure 4 is the rapid decrease in absorption as the energy of the photon increases. Also at a fixed energy σ_a increases much quicker than the atomic number Z. For our experiments $Z \propto \sigma_a^4$.

3 Apparatus

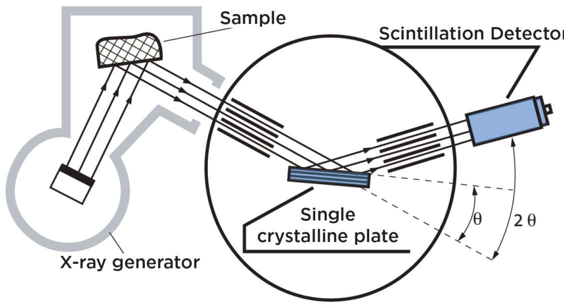

Figure 6: X-Ray Spectroscopy illustration

The x-rays are emitted from a molybdenum anode, Which then are incident on the mono-crystal, in our case sodium chloride is used. The sodium chloride disperses the x-rays into their spectrum and they are then detected by the counter tube. The spectrum recorded is sent the to computer and shown on the x-ray spectroscopy software for further analysis. also included the the experiment apparatus is the metal foils.

4 Procedure

4.1 Experiment 1

For this experiment the x ray spectrum was recorded for several different voltages, 20-35kv, in leaps of 35kv. A beta range was set from $3^{-}12$ with a $\Delta\beta$=0.1. The sodium chloride was mounted onto its stand. Then the spectrometer was calibrated in order to be set up at the correct angle for maximum intensity.
After the spectra was recorded, the minimum wavelength times the voltage was show to be a constant.
Finally the photon energies of k_{alpha} and k_{β} were measured and compared with the the previously calculated values.

4.2 Experiment 2

4.2.1 Experiment 2.1*K absorption edges*

The aims in the experiment are as follows: **i)** to determine the energy at the k absorption edge for zirconium, molybdenum, silver and indium **ii)** show that the $E_k \propto (Z - \sigma_k)^2$ **iii)** determine Rydberg constant R and the value of σ_k
. Also the transmittance was to be determined as a function of wavelength for each foil. Firstly the same setup as before was used, however this time the voltage stayed at 35keV. A beta range was set from $3^{-}12$ with a $\Delta\beta$=0.1. Now the spectrum was recorded for no foil, and again with each of the materials mentioned above as aa foil over the anode.

4.2.2 Experiment 2.2 *Mass attenuation Coefficient and Absorption Cross-Section*

The aims of this experiment are as follows; i) determine the mass attenuation coefficient $\frac{\mu}{\rho}$, for the foils as in experiment 2.1 ii) to find the dependence of σ_a on a Z wavelength.
In order to do this we will measure the attenuation at a fixed wavelength away from the edges. The same setup was used as in experiment 2.1. A constant wavelength to measure at was selected to be 41pm, this wavelength was selected as it was well below an edge but not such

that the intensity became too small. The beta angle was chosen from the wavelength based on Bragg's law wich in turn was 4.2 The following were set: $\Delta\ t = 20s$, $\Delta\ \beta = 0$

5 Results

5.1 Experiment 1

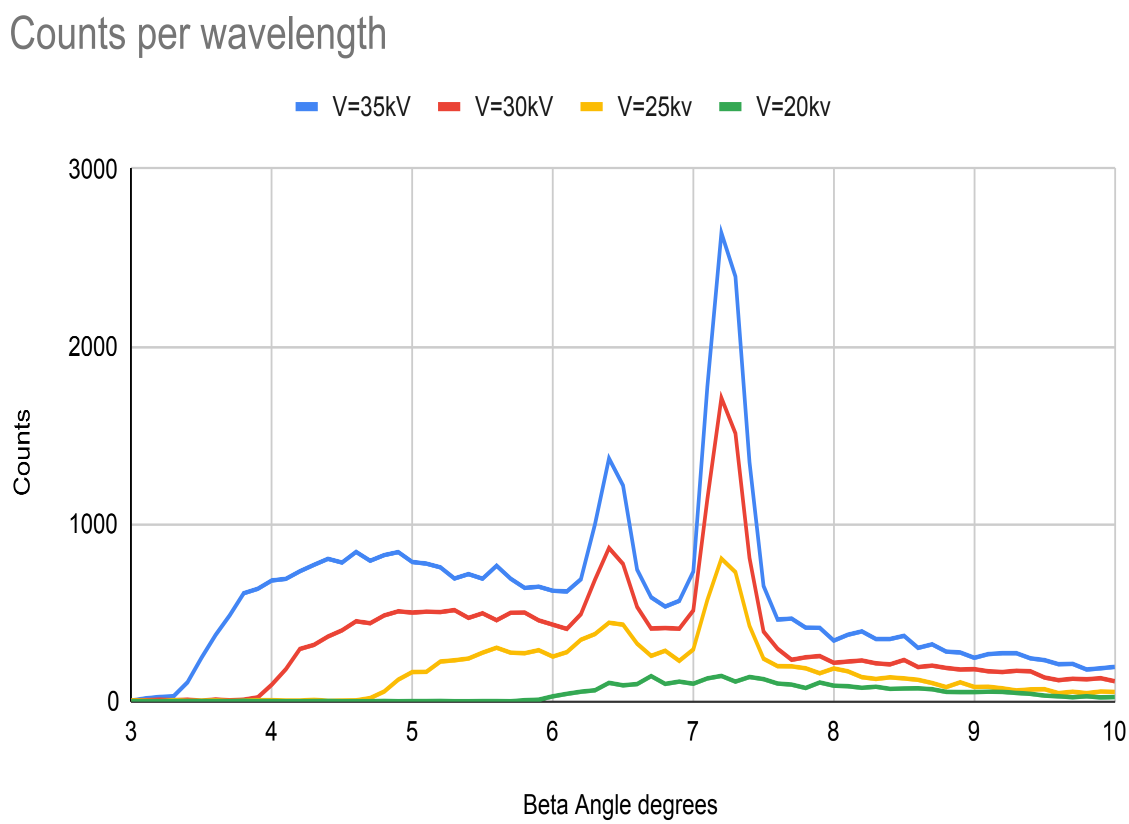

Figure 7: Counts per Wavelength molybdenum anode at various volts

Table 1: Experiment 1 Planck's Constant

V(kV)$\pm 0.05keV$	$\lambda\_min \pm 0.05(pm)$	V$\cdot\lambda_min \pm 0.0025(\mu meV)$	Planck's constant experimental /s	diff. w. Planck's constant /s
35	32.5	1.2	$6.39 \cdot 10^{-34} \pm 8.33 \cdot 10^{-36}$	$0.29 \cdot 10^{-34} \pm 8.33 \cdot 10^{-36}$
30	38.4	1.2	$6.39 \cdot 10^{-34} \pm 8.33 \cdot 10^{-36}$	$0.29 \cdot 10^{-34} \pm 8.33 \cdot 10^{-36}$
25	46.2	1.2	$6.39 \cdot 10^{-34} \pm 8.33 \cdot 10^{-36}$	$0.29 \cdot 10^{-34} \pm 8.33 \cdot 10^{-36}$
20	58	1.2	$6.39 \cdot 10^{-34} \pm 8.33 \cdot 10^{-36}$	$0.29 \cdot 10^{-34} \pm 8.33 \cdot 10^{-36}$

Table 2: Experiment 1:$K_{\alpha,\beta}$ wavelengths and energies

V(kV)	$K_\alpha\lambda$(pm)	$K_\beta\lambda$(pm)	$K_{E\alpha}$keV	$K_{E\beta}$keV	diff w. calculated($K_{E\alpha}$) (keV)	diff w. calculated ($K_{E\beta}$)(keV)
35	70.7±0.05	62.9±0.05	17.58±0.013	19.75±0.009	0.38±0.013	0.65±0.009
30	70.7±0.05	62.9±0.05	17.58±0.013	19.75±0.009	0.38±0.013	0.65±0.009
25	70.7±0.05	62.9±0.05	17.58±0.013	19.75±0.009	0.38±0.013	0.65±0.009
20	70.7±0.05	62.9±0.05	17.58±0.013	19.75±0.009	0.38±0.013	0.65±0.009

5.2 Experiment 2

5.2.1 Experiment 2.1

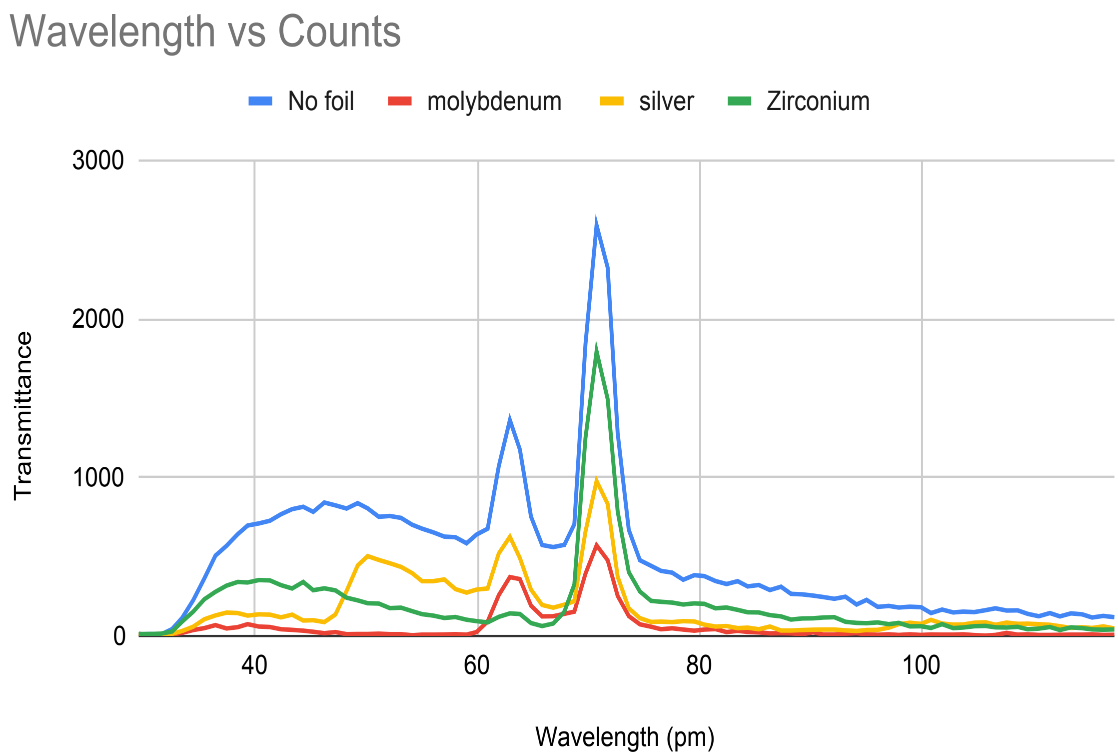

Figure 8: Experiment 2.1 wavelength vs counts for various foils

Disclaimer: Due to error in recording indium reading is omitted.

Table 3: Experiment 2.1 Results Table

Covering	$K_{edge}T$	K edge λ(pm)	$K_{edge}Energy$(keV)	$K_{edge}Energy$(J)	σ_k (b/N_a)	$h \cdot c \cdot (Z - \sigma\_k)^2$	Z
Zirconium	0.47±0.08	68.7±0.05	18.06±0.11	2.89e-15±0.04	206.96±39.34	5.54e-21±2.307e-21	40
Molybdenum	0.22±0.08	60.9±0.05	20.37±0.13	3.26e-15±0.02	162.2±25.76	1.86e-22±8.60e-22	42
Silver	0.31±0.08	48.2±0.05	25.74±0.15	4.12e-15±0.75	239.98±41.93	7.067e-21±2.80e-21	47

From table 3 one can see the k edge energies.

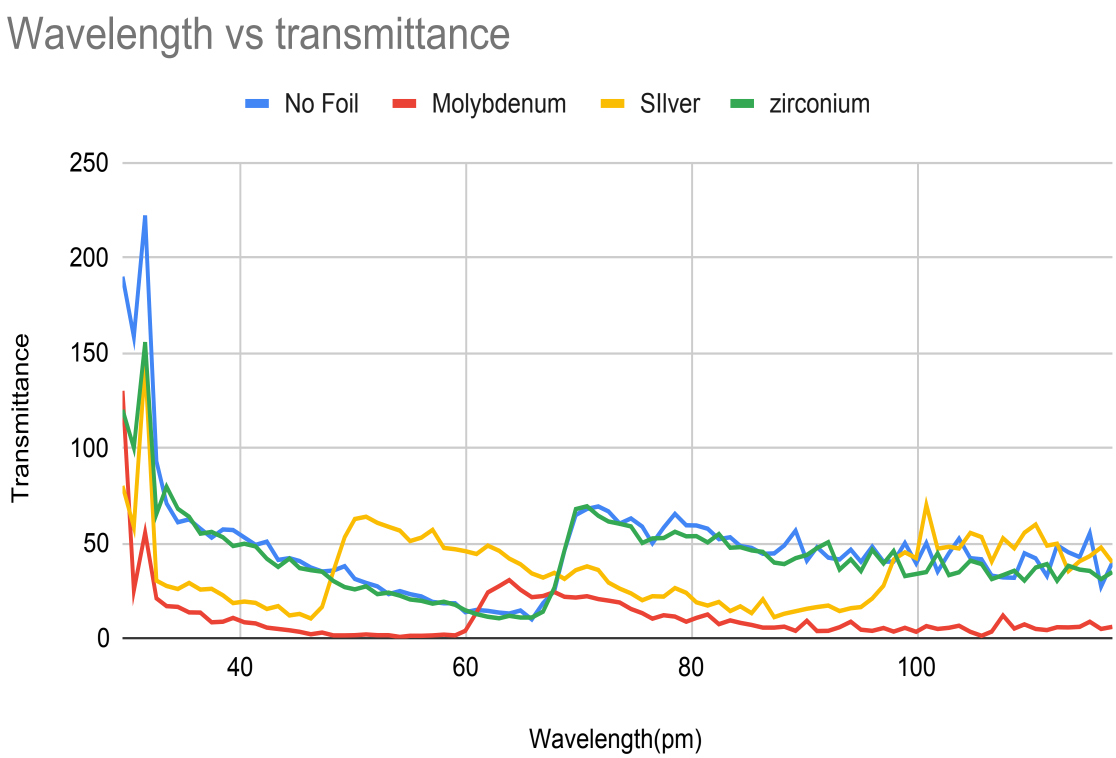

Figure 9: Experiment 2.1 wavelength vs transmittance for various foils

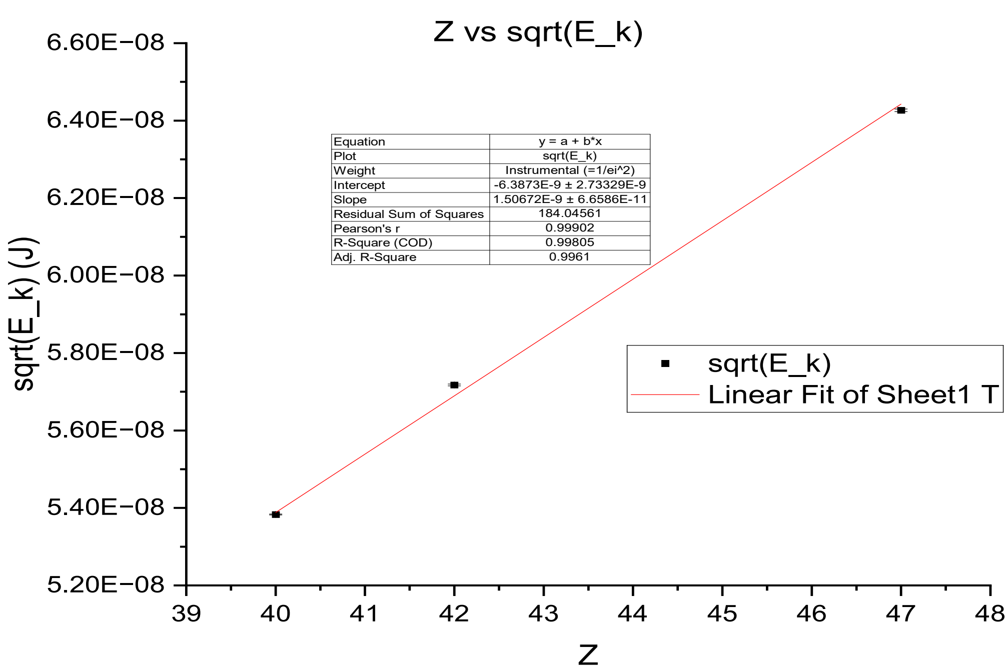

Figure 10: Experiment 2.1 Z vs $\sqrt{E_k}$

This plot Enables one to calculate R as $\frac{m^2}{hc} = R \approx 1.14 \cdot 10^7/m \pm 1.03 \cdot 10^6/m$, with m being the slope.

5.2.2 Experiment 2.2

Table 4: Experiment 2.2 Results table

Filter	Mean count rate \s	R/R0=T	$\sigma\_a(b)$)	Z	Density (kg/m^2)	Thickness(m)	$\frac{\mu}{\rho}(m^2/kg)$
No filter	224.5±0.05	1	-	-	-	-	-
Zr	103.7±0.05	0.461±2.2e-4	216.55±0.13	40	6507	0.05·10-3	2.38±0.001
Mo	13.85±0.05	0.0616±2.2e-4	261.44±0.03	42	10222	0.1·10-3	2.72±0.0004
Ag	40.5±0.05	0.18±2.2e-4	351.87±0.25	47	10500	0.05·10-3	3.26±0.002
In	1.3±0.05	0.0057±2.2e-4	270.46±1.98	49	7290	0.3·10-3	2.36±0.017

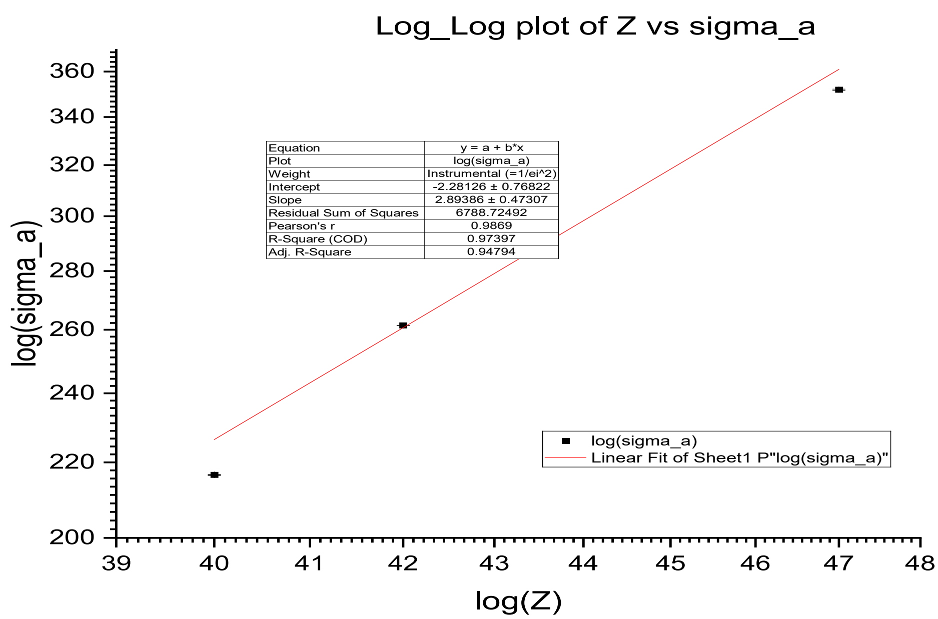

Figure 11: Experiment 2.2 log log plot for Z vs σ_a

(Indium left out of plot as it was found to be an outlier)

From Figure 11 one can determine that $\sigma_a \propto Z^{2.84 \pm 0.47}$

6 Error Analysis

The error analysis for the functions of resulting variables was as follows :

$$\Delta f = \frac{\partial f}{\partial x}\Delta + \frac{\partial f}{\partial y} \tag{14}$$

for f being a function of x and y.

7 Conclusion

The x-ray spectra was successfully recorded at accelerating voltages of 20-35 keV. $\lambda_{min} = const$ was found to be $1.2\mu meV \pm 0.0025\mu meV$. The calculated Planck's constant was found to be equal to $6.39{\cdot}10^{-34}/s \pm 8.33{\cdot}10^{-34}/s$ which had a difference of $0.29{\cdot}10^{-34} \pm 8.33{\cdot}10^{-34}/s$. The photon energies of the alpha and beta K lines in the characteristic spectrum of molybdenum were found to be 17.58±0.013 keV and 19.75±0.009 keV respectively, which varied by 0.38±0.013 keV and 0.65±0.009 keV from the calculated values in the theory section respectively. The energy at the K absorption edge for zirconium, molybdenum and silver were found to be 18.06 keV±0.11keV, 20.37 keV±0.13keV and 25.74 keV±0.15keV respectively. The Rydberg constant was determined to be $R \approx 1.14 \cdot 10^7/m \pm 1.03 \cdot 10^7/m$. The mass attenuation coefficients of the foils were calculated to be $2.38m^2/kg \pm 0.001m^2/kg$ for Zr, $2.73m^2/hg \pm 0.0004m^2/kg$ for Mo, $40.5m^2/kg \pm 0.002m^2/kg$ for Ag, and $2.36m^2/kg \pm 0.017m^2/kg$ for indium. The absorption cross sections of the foils at a fixed wavelength were calculated and found $\sigma_a = 216.55(b/N_a) \pm 0.13(b/N_a)$ for zirconium, $\sigma_a = 261.44(b/N_a) \pm 0.03(b/N_a)$ for Molybdenum, $\sigma_a = 351.87(b/N_a) \pm 0.25(b/N_a)$ for Silver, and $\sigma_a = 270.46(b/N_a) \pm 1.98(b/N_a)$ for Indium. It was found that $\sigma_a \propto Z^{2.84\pm0.47}$, which lies outside of the expected $\sigma_a \propto Z^4$.

References

[1] Lawrence Berkeley National Laboratory. *X-ray Data Table*. Available at: `https://xdb.lbl.gov/Section1/Table_1-2.pdf` [Accessed 23 Feb. 2025]. n.d.

[2] Unacademy. *Continuous X-rays*. Available at: `https://unacademy.com/content/jee/study-material/chemistry/continuous-x-rays/` [Accessed 23 Feb. 2025]. n.d.

[3] James H. Wittke. *The Origin of Characteristic X-rays*. Archived from the original on 9 July 2013. Retrieved 18 June 2013. 2013. URL: `https://web.archive.org/web/20130709000000/http://example.com`.

List of Figures