Lab report 6: X-ray Absorption Spectroscopy

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Contents

List of Figures

List of Tables

Abstract

This lab dealt with various characteristics of the X-Ray spectrum and how it interacted with a variety of materials. The first experiment required testing the effect of different accelerating voltages, U, on the spectrum of Molybdenum. It was found that as energy decreased, so too did the size of the peaks until at 20KeV, the α and β peaks had all but disappeared. It was shown that the accelerating voltage multiplied by the minimum wavelength at which a current could be read returned a constant $U\lambda_{min}$, from which we could verify Planck's constant to be $(6.167 \pm 0.231) \times 10^{-34} Js$, very close to the literature value of $6.626 \times 10^{-34} Js$. The energies of the peaks $E_{K\beta}$ and $E_K\alpha$ were calculated from the data obtained($19.53 \pm 0.3 KeV$ and $17.26 \pm 0.3 KeV$ respectively) and then verified using the modified bohr model to obtain theoretical values(20.26KeV and 17.28KeV).

The second experiment dealt with various aspects of X-ray attenuation, the first of which tested being the K absorption edge for Zr, Mo, Ag and In foils. The edge wavlengths for each foil were found and tabulated. From these it was possible to verify the Rydberg constant which was calculated as $(1.193 \pm 0.115) \times 10^7 m^{-1}$, within experimental error of the literature value of $1.097 \times 10^7 m^{-1}$. The shell screening parameter could also be deduced to be $\sigma_K = 33.66 \pm 3.6$.

The final part of this experiment involved measuring the mass attenuation coefficient μ/ρ and absorption cross section σ_a for various metal foils : Al, Fe, Cu and Zr. T, the transmission was measured and from this(along with values supplied), the desired values of μ/ρ and σ_a were calculated and tabulated. To verify the veracity of these values, it was necessary to verify the law σ_a^p where the literature value of p is 4. The obtained value was 3.45 ± 0.12 which is reasonably in line with expectation.

note: references to the bibliography are denoted by |**x**| where x corresponds to the index number of the bibliography entry.

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I. Theory

I. The X-ray spectrum

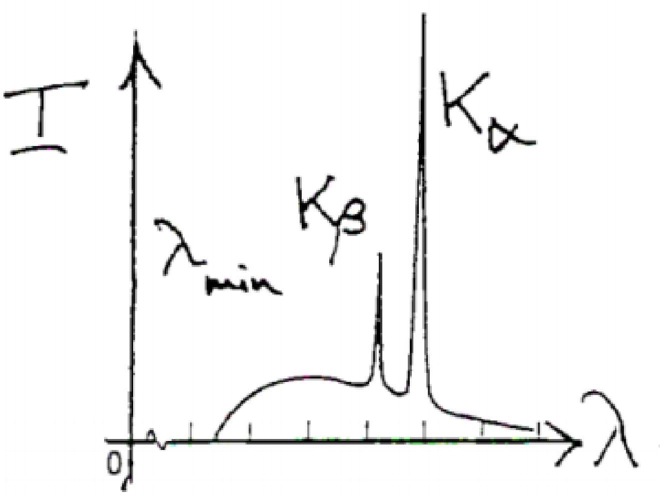

Figure 1: Typical X-ray spectrum

The typical x-ray spectrum appears as above with β radiation appearing at a lower wavelength due to its higher energy(i.e $E = \frac{hc}{\lambda}$). It then makes sense that the α radiation is further ahead. This spectrum arise from transitions of electrons between the inner shells of the target atoms. An incident electron may knock an electron out of one of the inner shells of a target atom.

The *characteristic spectrum* arises from transitions of electrons between the inner shells of the target atoms. An incident electron may knock an electron out of one of the inner shells of a target atom. Suppose one of the two K shell electrons is removed. As shown in figure A2.2 this vacancy can then be filled by an electron falling in from one of the outer shells such as L, M … with the emission of a photon whose energy, except for the lightest elements, is in the x-ray region of the spectrum.

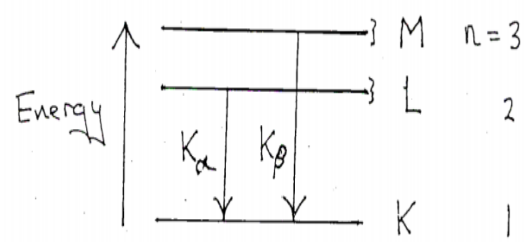

Fig A 2.2

The terms Kα , Kβ refer to transitions from L → K and M → K respectively. An estimate of the corresponding photon energies $E_{K\alpha}$, $E_{K\beta}$, may be obtained using a modified version of the Bohr model that includes the effect of the electrons on screening the nuclear charge Z e in a nucleus of atomic number Z . This model gives the binding energy of the electrons in the shell with quantum number n as

$$E_n = - R h c Z^2_{eff} / n^2$$

Figure 2: lab manual explanation and diagram

It is then possible to calculate $E_{k\alpha}$ and $E_{k\beta}$ energies using this revised bohr model. A transition from the third energy level to the first energy level as is the case for β radiation is like so

$$E_{K\beta} = \frac{-RhcZ_{eff}^2}{9} - (-\frac{RhcZ_{eff}^2}{1})$$

$$E_{K\beta} = \frac{8RhcZ_{eff}^2}{9}$$

for the case of α radiation, we have a transition from the second to the first energy level

$$E_{K\alpha} = -\frac{RhcZ_{eff}^2}{4} - (-\frac{RhcZ_{eff}^2}{1})$$

$$E_{K\alpha} = \frac{3RhcZ_{eff}^2}{4}$$

The value of Z_{eff} is normally given by $Z - \sigma_m$, but for an electron falling down towards the K level, we can set $\sigma_m = 1$, since there are only two electrons in the first orbital($1S^2$), so if one of these is ejected, only one remains to screen the nucleus.

II. The K absorption edge

The absorption edge occurs when enough energy is supplied to suddenly remove an electron from the K-shell, thereby causing a sudden change in absorption like so

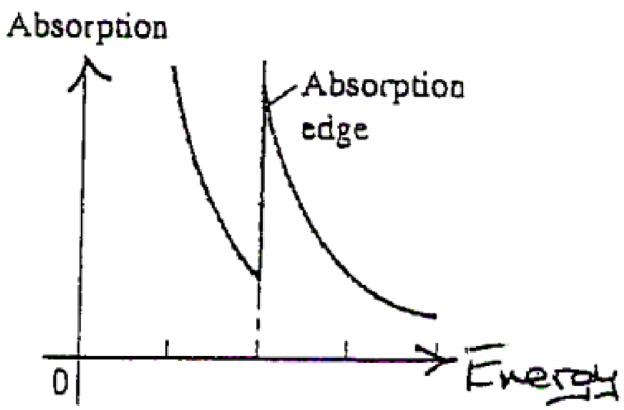

Figure 3: The K-absorption edge example

III. The mass attenuation coefficient and absorption cross section

The mass attenuation coefficient is the linear attenuation coefficient divided by the density of the material $\mu.\rho$. This refers to how easily a material can be penetrated by energy or radiation. The total cross section σ is the sum of scattering and absorption cross sections $\sigma_s + \sigma_a$. It is then a simple manner to rearrange for σ_a.

II. Experimental Procedure

I. Experiment 1: The X-Ray Spectrum

This experiment involved measuring the X-ray spectrum of Molybdenum and making subsequent calculations of which I will go through. Since we are dealing with X-rays which carry radiation, the experiment was carried out in a somewhat bulky box to shield the researcher. The $NaCl$ crystal was set up like so

Figure 4: Complete shielded X-ray spectrum(Mo) apparatus(German-make:Leybold shop)

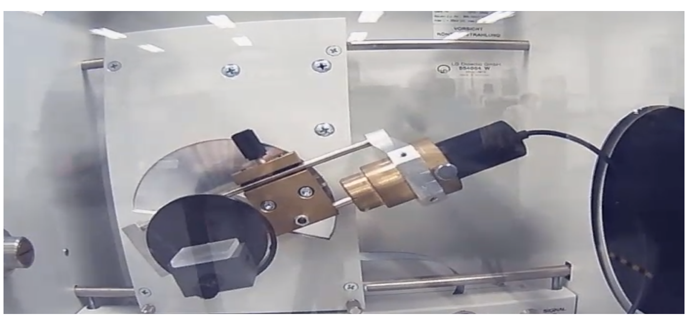

Figure 5: NaCl crystal mounted on support

The NaCl sample was flush against the walls of the holder to avoid any slight angles. The angle of the apparatus can be changed via computer or manually via a knob on the box itself as one can no doubt surmise from the picture above.

Now that the technical details are out of the way, the machine was hooked up to a computer to print the resulting spectrum. The COUPLED setting was used and the following initial parameters were set ;U=35KeV, $\Delta\beta^\circ = 0.1^\circ$, $\Delta t = 5sec$,$I = 1mA$, $\beta_i^\circ = 3^\circ$ and $\beta_f^\circ = 10^\circ$. It is important not to confuse the supply current of 1mA with the current being read at various angles which is more like an intensity, the number of electrons per unit time being read as opposed to electricity through a wire.

Once this was done I pressed SCAN and saving the resulting spectrum. I repeated this experiment for voltage values of U=30KeV, U=25KeV and U=20KeV, taking care to overlay these on top of each other. This concludes the data acquisition which leads me to the data analysis of this experiment.

For future reference, any angular measurement made on the x axis can be converted to a wavelength value λ using the equation $n\lambda = 2dSin(\beta)$ where n=1, 2d=536pm for NaCl and β is our angle of interest.

The three main areas of interest our spectra were λ_{min}, the minimum wavelength at which a current could be read, $\lambda_{K\alpha}$, the wavlength at which alpha radiation occurs(this is the peak with the higher wavelength, meaning a lower energy since beta radiation is stronger) and $\lambda_{K\beta}$, the peak at the lower wavelength. The energy returned by these respective wavelengths can be calculated using the equation

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

no surprises there. The values of λ_{min} can be utilised to verify Planck's constant according to the following equation

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

Since the right handside is nothing but a series of constants, so too will the left hand side return a constant for our varying values of U and λ_{min}. This being the case, we can average the values of $U\lambda_{min}$ for each reading and rearrange to find h like such

$$h = \frac{U\lambda_{min} \times e}{c}$$

This value was calculated with the proper dimensional analysis carried out.

As further proof of the veracity of these experiments I was also able to calculate the theoretical values of $E_{K\alpha}$ and $E_{K\beta}$ and compare these to the obtained values. These were calculated using the following equations, the derivation of which is covered in the theory section

$$E_{K\alpha} = \frac{3RhcZ^2}{4} \quad and \quad E_{K\beta} = \frac{8RhcZ^2}{9}$$

where R is the Rydberg constant, h and c have their usual meanings, and Z is the effective atomic number of the NaCl crystal(14).

II. Experiment 2:X-Ray attenuation

II.1 K - Absorption Edge

The aim of this experiment was to find the K_{edge} for multiple metal foils(Zr,Mo,Ag and In) and from this to extrapolate several useful relations which I shall go through. A scan was undertaken like before with the only change in parameters being that the beginning and starting angle for this run were $\beta_i^\circ = 3^\circ$ and $\beta_f^\circ = 12^\circ$. The x axis scale was changed to a λ scale by setting the device to read for NaCl. The Y axis was changed to transmission(R/R0) simply by clicking on the transmission button.

The scan was conducted for a Zr foil and the resulting distribution was printed onto the screen. This was repeated for foils of Mo, Ag and In. It should be noted here that the key feature we are looking for is the k edge, the sudden "cliff" of sorts in the data that is very evident to the untrained eye. The position of these were noted for each metal foil. The energy corresponding to each of these wavelengths $E = hc/\lambda$ was calculated and tabulated. Now it is time for some simple graphical analysis. The energy E_K which we calculated is also given by the relation

$$E_K = Rhc(z - \sigma_K)^2$$

$$\sqrt{E_K} = \sqrt{Rhc}(Z) - \sqrt{Rhc}(\sigma_K)$$

$$\sqrt{\frac{E_K}{hc}} = \sqrt{R}(Z) - \sqrt{R}(\sigma_K)$$

$$\sqrt{\frac{1}{\lambda}} = \sqrt{R}(Z) - \sqrt{R}(\sigma_K)$$

This is in the form

$$Y = mx + c$$

Therefore, if we plot Z(atomic number) on the x axis and $\sqrt{1/\lambda}$ on the y axis, the slope must equal $\sqrt{R}$ which we need only square to return us the verification of the Rydberg constant. Once we calculated $\sqrt{R}$, we then divided the y intercept c by this number to return σ_K, the desired value.

II.2 Mass attenuation coefficient and absorption cross-section

To determine the mass attenuation coefficient μ and absorption cross-section σ_a for Al, Fe, Cu and Zr at a fixed wavelength away from the absorption edges. The wavelength was chosen as 41pm which returned an angle of $\beta^\circ = 4.2^\circ$.

This ideal angle was set manually using the dial on the device. The initial R_0 was found by setting $\Delta\beta^\circ = 0^\circ$, $\Delta t = 20s$ and pressing scan. This basically means that we took a scan without changing the angle for 20 seconds. To find the mean count rate I pressed replay on the apparatus, returning the mean count rate for the source. This exact procedure was repeated for each varying foil and the transmission was calculated by dividing the count rates of each respective foil by the source count rate $R/R_0 = I/I_0$. This is handy since we also have the relation

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

$$\frac{I}{I_0} = e^{-\mu x}$$

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

where x is the thickness of the foil, μ is the linear attenuation coefficient of the material and T is the transmission. Thus one can calculate the mass attenuation coefficient for each sample by taking μ/ρ where ρ is supplied to us. Getting at σ_a requires a bit more work. We know that the total absorption cross section is the sum of scattering and absorption

$$\sigma = \sigma_a + \sigma_s$$

$$\sigma_a = \sigma - \sigma_s$$

$$\sigma_a = \frac{A\mu}{N_A\rho} - \frac{0.02A}{N_A}$$

where N_A is Avogadro's number, ρ is the density of the sample, A is the atomic weight of the material, and μ is the linear attenuation coefficient. All of these values are either supplied or calculated at this point thus we can simply plug the values in to return σ_a.

It is stated in the lab manual that $\sigma_a \propto Z^p$ where p is just a constant power law. Since the only value not known here is p, we can refer once again to graphical analysis, plotting $ln(\sigma_a)$ vs $ln(Z)$ to return a straight line where the corresponding slope is the number p as required. This concludes the experimental procedure of this experiment.

III. Results and Discussion

I. Experiment 1: The X-Ray Spectrum

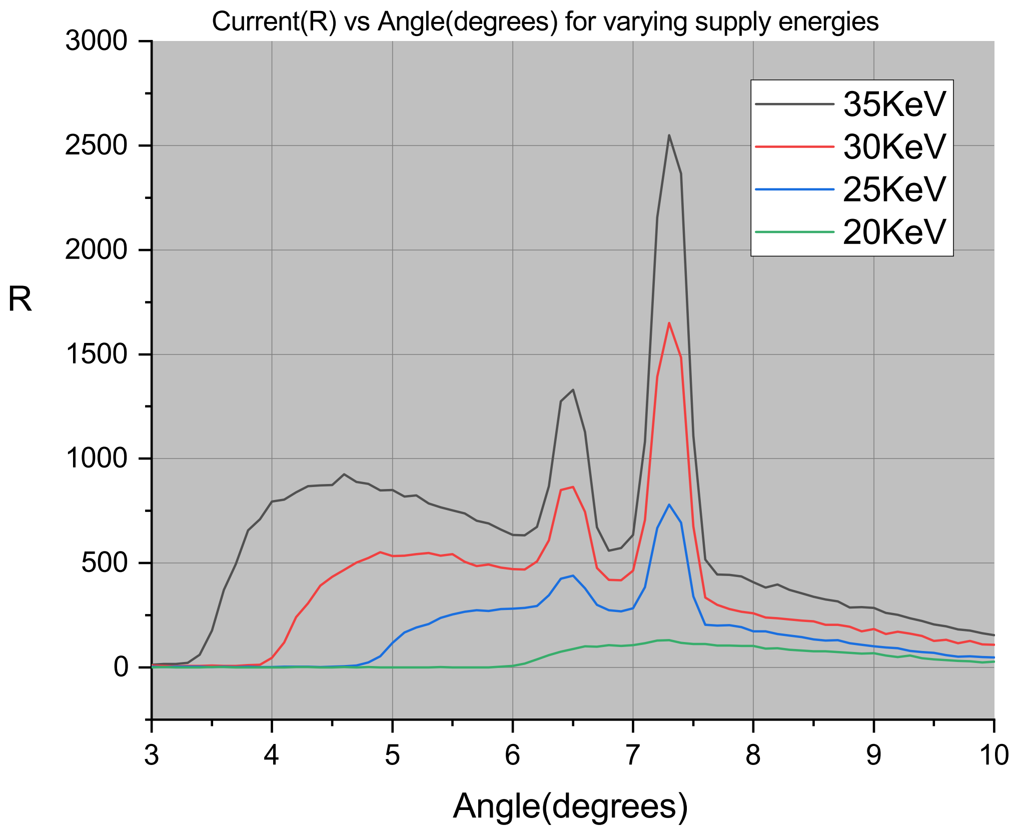

Figure 6: X-Ray emission spectrum

X Ray Spectrum				
Energy(KeV)	35	30	25	20
$\lambda_{min}(pm)$	32.3 ± 1.95	38.10 ± 1.95	46.5 ± 1.95	59.1 ± 1.95
$U\lambda_{min}$(KeV.pm)	1130.5 ± 68.2	1143.2 ± 58	1175 ± 48.67	1180.1 ± 39
$\lambda_\beta(pm)$	64.14 ± 1.95	63.35 ± 1.95	63.44 ± 1.95	————
$\lambda_\alpha(pm)$	73.22 ± 1.95	71.57 ± 1.95	71.44 ± 1.95	————
$E_{max}(KeV)$	38.4 ± 2.32	32.60 ± 1.66	26.7 ± 1.12	21.04 ± 0.7
$E_\beta(KeV)$	19.37 ± 0.2	19.61 ± 0.3	19.6 ± 0.3	————
$E_\alpha(KeV)$	16.98 ± 0.51	17.4 ± 0.19	17.39 ± 0.19	————

Table 1: The X-Ray Spectrum

While the $U\lambda_{min}$ values show some slight deviation from one another, they are all within experimental error of each other respectively. this in units of KeV.pm.
It is then possible to take an average of these values and utilise them to calculate Planck's constant like such

$$\frac{(1130.5 \pm 68.2)KeV.pm + (1143.2 \pm 58) + (1175 \pm 8.75)KeV.pm + (1180.1 \pm 39)Kev.pm}{4} = 1153.90 \pm 43.49Kev.pm$$

Converting this to proper units we have

$$(2307.8 \pm 43.39)Kev.pm \times \frac{10^3 eV}{KeV} \times \frac{1m}{10^{12}pm} \times \frac{1.6 \times 10^{-19}J}{eV} = (1.85 \pm 0.07) \times 10^{-25}J.m$$

It is known that the experiment obeys the equation

$$U \times \lambda_{min} = \frac{hc}{e}$$

Rearranging for h, Planck's constant and keeping in mind that we have already multiplied by e in converting to proper units we have

$$h = \frac{U \times \lambda_{min}}{c} = \frac{(1.85 \pm 0.07) \times 10^{-25}J.m}{3 \times 10^8 ms^{-1}}$$

$$h = (6.167 \pm 0.231) \times 10^{-34}Js$$

The accepted literature value for Planck's constant is $6.626 \times 10^{-34}Js$. This is very close to being within experimental error and dealing with small numbers like these, it is not uncommon that we would have a very slight error, thus it isn't too egregious to say that we have indeed verified Planck's constant.

The theoretical values for $E_{K\alpha}$ and $E_{K\beta}$ were calculated using the respective formulae

$$E_{K\alpha} = \frac{3RhcZ^2}{4} = \frac{3 \times 1.097 \times 10^7 m^{-1} \times 6.626 \times 10^{-34}Js \times 3 \times 10^8 ms^{-1}(13)^2}{4} \times \frac{1eV}{1.6 \times 10^{-19}J} \times \frac{10^{-3}KeV}{eV} = 17.28KeV$$

$$E_{K\beta} = \frac{8RhcZ^2}{9} = \frac{8 \times 1.097 \times 10^7 m^{-1} \times 6.626 \times 10^{-34}Js \times 3 \times 10^8 ms^{-1} \times 13^2}{9} \times \frac{1eV}{1.6 \times 10^{-19}J} \times \frac{10^{-3}KeV}{eV} = 20.26KeV$$

These compare rather nicely with the obtained values which averaged out to be $E_{K\beta} = (19.53 \pm 0.3)KeV$, and $E_{K\alpha} = (17.26 \pm 0.3)KeV$

II. Experiment 2:X-Ray Attenuation

II.1 Absorption edge

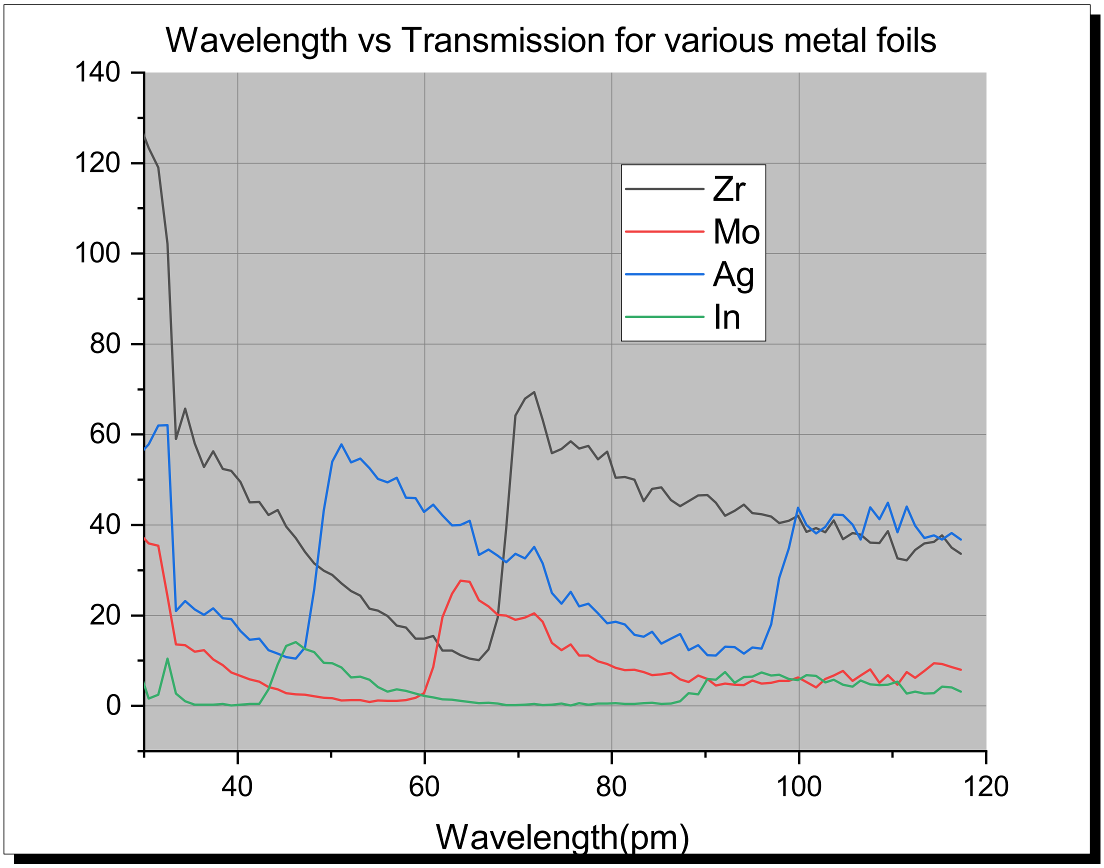

Figure 7: Transmission spectrum for various foils

Transmission Spectrum				
	Zr	Mo	Ag	In
Z	40	42	47	49
$\lambda_K(pm)$	70 ± 1.95	63 ± 1.95	50 ± 1.95	42 ± 1.95
$E_K(KeV)$	17.75 ± 0.03	19.72 ± 0.61	24.85 ± 0.97	29.58 ± 1.37
$\sqrt{\frac{1}{\lambda_k}}(m^{-1/2}) \times 10^5$	(1.20 ± 0.03)	(1.26 ± 0.04)	(1.41 ± 0.06)	(1.54 ± 0.07)

Table 2: Transmission spectrum for various foils- Relevant data

A graph of $\sqrt{\frac{1}{\lambda_k}} vsZ$ was constructed as discussed in the experimental method(see next page)

As one can see from the graph above the slope was returned as $(0.03453 \pm 0.00334) \times 10^5 m^{-1/2}$. Referring back to the experimental method, our line is the form y=mx+c where m=$\sqrt{R}$ and c=$\sqrt{R}\sigma_K$ like such

$$\sqrt{\frac{1}{\lambda_K}} = \sqrt{R}Z - \sqrt{R}\sigma_K$$

So our slope should equal $\sqrt{R}$, therefore $R = (1.193 \pm 0.115) \times 10^7 m^{-1}$. The accepted literature value is $1.097 \times 10^7 m^{-1}$ which is within experimental error of the obtained value. From this, it is then possible to calculate σ_K, since we know the y intercept,c, must equal $\sqrt{R}\sigma_K$.

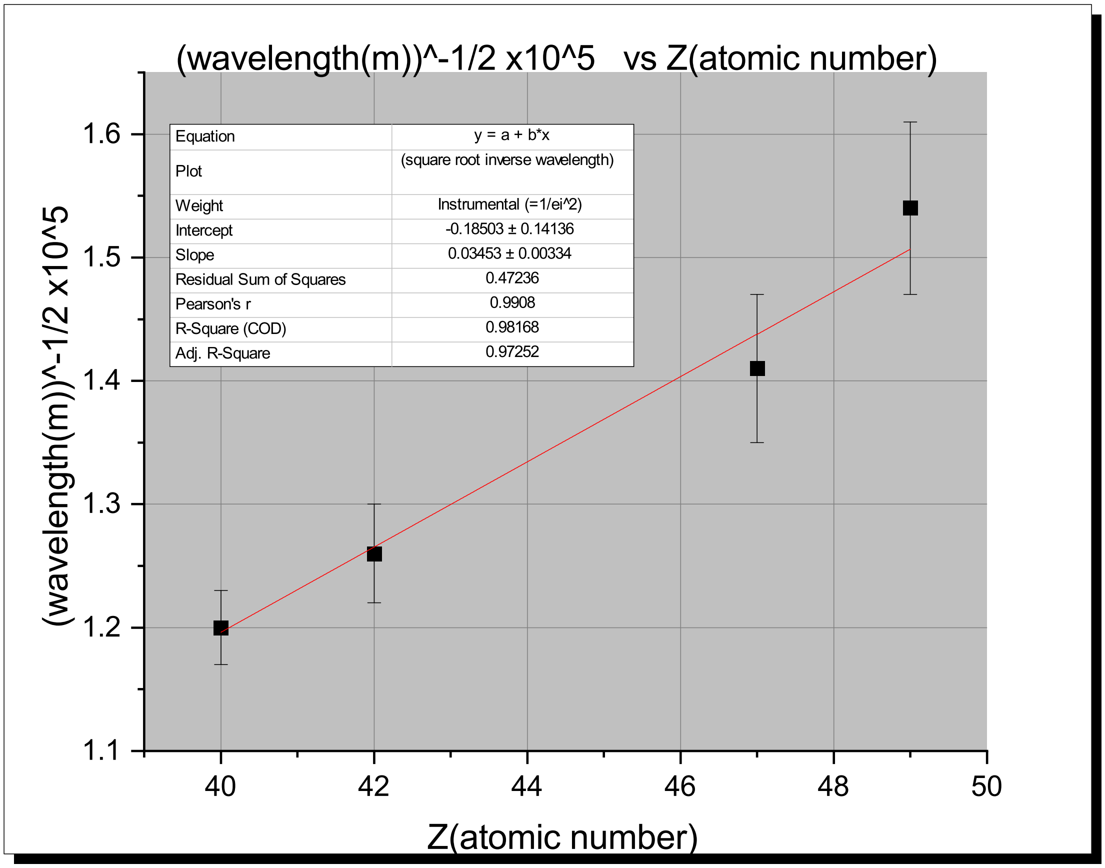

Figure 8: $\lambda^{-1/2}$ vs Z(atomic number)

The y intercept was calculated to be $(1.1625 \pm 0.00334) \times 10^5 m^{-1/2}$. We need only divide this by the $\sqrt{R}$ value to obtain σ_K

$$\sigma_K = \frac{(1.1625 \pm 0.00334) m^{-1/2}}{(0.03453 \pm 0.00334) \times 10^5 m^{-1/2}} = 33.66 \pm 3.6$$

. This is a dimensionless number since it is merely a ratio of the screening at the k edge.

II.2 Mass attenuation and absorption cross-section

Mass attenuation and absorption cross-section				
	Al	Fe	Cu	Zr
Z	13	26	29	40
Initial counts(s^{-1})	502.8 ± 22.4	502.8 ± 22.4	502.8 ± 22.4	502.8 ± 22.4
Counts(s^{-1})	389.6±19.73	19.75±4.4	255.9 ± 15	222.1 ± 14.9
T	0.77 ± 0.04	0.04 ± 0.01	0.51 ± 0.03	0.44 ± 0.03
x(mm)	0.5	0.5	0.07	0.05
A	26.98	55.84	63.54	91.22
μ	522.73 ± 27.16	6437.8 ± 446.34	9619.2 ± 565.84	16420 ± 1119.6
$\rho(kgm^{-3})$	2698	7873	8933	6507
$\frac{\mu}{\rho}(m^2kg^{-1})$	0.19 ± 0.01	0.82 ± 0.05	1.08 ± 0.06	2.52 ± 0.2
$\sigma_s(m^2) \times 10^{-22}$	0.089933	2.6243	2.9777	2.1690
$\sigma(m^2) \times 10^{-20}$	0.0854 ± 0.0045	1.076 ± 0.065	1.4171 ± 0.0787	3.3067 ± 0.2624
$\sigma_a(m^2) \times 10^{-20}$	0.08454 ± 0.0045	1.0498 ± 0.065	1.3873 ± 0.0787	3.2850 ± 0.2624

Table 3: Mass attenuation and absorption cross section relevant data

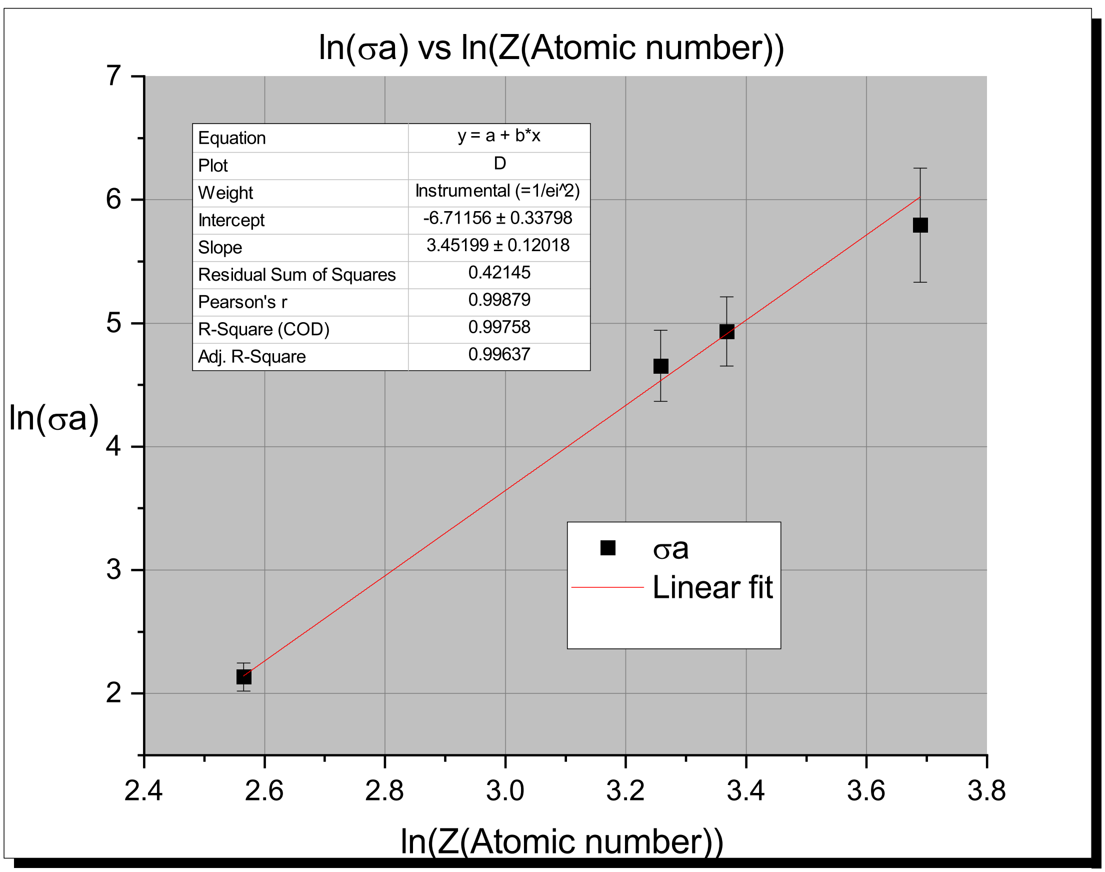

Figure 9: $ln(\sigma_a)$ vs $ln(Z)$: To determine P from the linear fit of $ln(\sigma_a) = Pln(Z) + C$

The value of P is returned as 3.45199 ± 0.12019 or 3.45 ± 0.12. The literature value for the P value given is as 4 so the closest we can get with the experimental error here is 3.57. This is somewhat off the expectation but not immensely so. This could be due to leaving the samples in for only 20 seconds, a longer time might have yielded more accuracy but I think this reasonably satisfies the relation $\sigma_a \propto Z^4$.

IV. Conclusion

In conclusion the photon energies of the α and β lines of the molybdenum x-ray spectrum were measured and planck's constant was measured as

$$h = (6.167 \pm 0.231) \times 10^{-34} Js$$

which is very close to the literature value of $6.626 \times 10^{-34} Js$. The theoretical values of the α energy and the β energy were in agreement with the experimental values.

The Rydberg constant was measured to be $R = (1.193 \pm 0.115) \times 10^{7} m^{-1}$ which is within experimental error of the literature value $1.097 \times 10^{7} m^{-1}$. The shell screening parameter,σ_k, was found to be

$$33.66 \pm 3.6$$

. The values of μ and σ_a were tabulated and it was observed that a relation existed between the atomic number Z and the absorption cross section according to the following ;$\sigma_a \propto Z^p$ where p was measured as 3.45 ± 0.12, the accepted value being 4 which is reasonably close to the obtained value.

References

[1] TrinityCollegeDublin/Schoolofphysics/JSlabs[*"X-ray Absorption Spectroscopy" 2013*]