

# **Experiment 10**

## **X-Ray Diffraction**

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10:00 A.M - 1.00 P.M

## Introduction:

This experiment involves the bombardment of salt crystal samples with X-rays which results in a diffraction that can be recorded and graphed. The graph which is produced involves two main features of the experiment. The large peak values  $K_{Alpha}$  and  $K_{Beta}$  are based on Electron hole's while the rest of the graph is based on 'Bremsstrahlung'. When a free electron strikes an s orbital electron and knocks it out of the atom, an electron hole is created within the atom and a p orbital electron drops down to fill the newly created hole giving off energy. This creates the peaks in our graph, which will help us find the wavelength of the X-ray beam, we need to use this wavelength in the Bragg's equation to find the distance between the lattice spaces in the salt crystal samples.

## Method:

In order to produce the graphs required to carry out this experiment, two salt crystals NaCl and LiF were placed in a Leybold Didactic x-ray diffraction apparatus. The machine allows us to adjust certain parameters such as the high voltage, emission current, how many seconds to measure each angle and by what interval to change the angle. Once we have produced an adequate graph with at least first and second order peaks, we can then calculate the average  $\lambda K_{\alpha}$  and  $\lambda K_{\beta}$ , finally we sub the order  $n$ , wavelength  $\lambda$  and angle  $\theta$  into the braggs equation:

$$n \lambda = 2 d \sin \theta \quad (1)$$

Our goal being to ultimately obtain a value for 'd' which is the space between two unlike atoms in a crystal lattice. Such as Sodium and Chlorine or Lithium and Fluoride.

## Results:

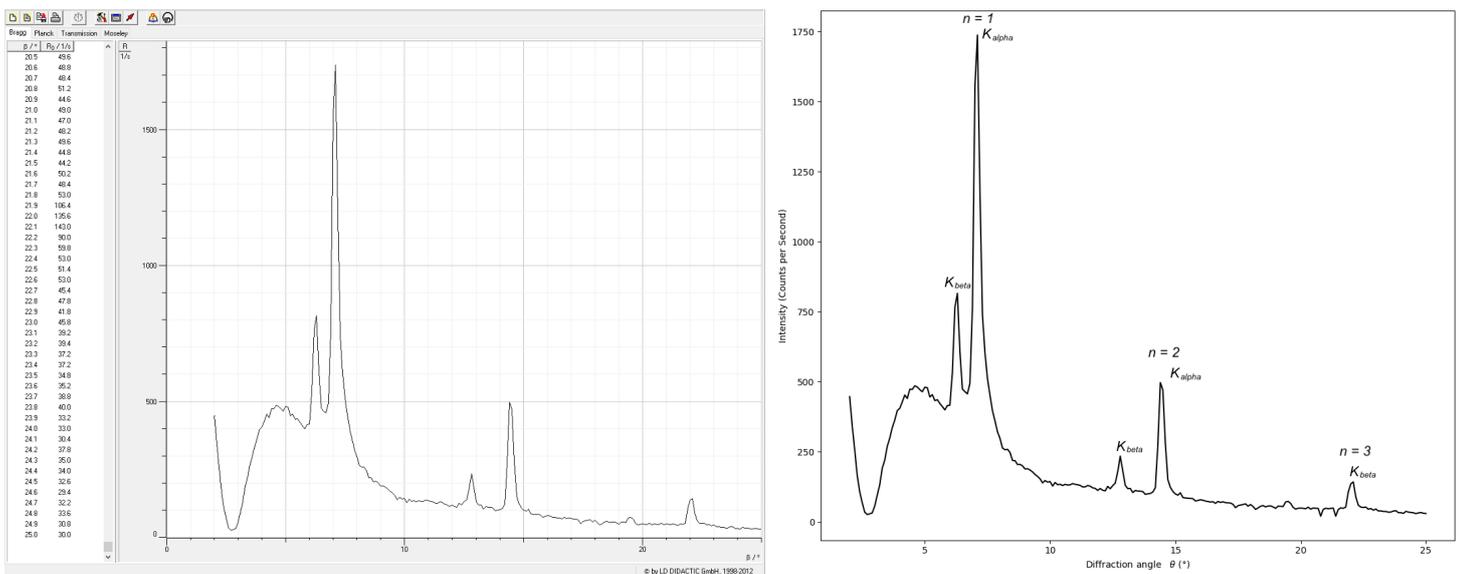


Figure 1: Graph of NaCl Diffraction

## NaCl Diffraction Peak Values

Values of  $n = 1$

	Diffraction Angles $\theta^\circ$	Intensity ( $R_0 \text{ s}^{-1}$ )
$K_{beta}$	6.3	815.8
$K_{alpha}$	7.3	1737.6

Values of  $n = 2$

	Diffraction Angles $\theta^\circ$	Intensity ( $R_0 \text{ s}^{-1}$ )
$K_{beta}$	12.8	234.8
$K_{alpha}$	14.4	497.4

Values of  $n = 3$

	Diffraction Angles $\theta^\circ$	Intensity ( $R_0 \text{ s}^{-1}$ )
$K_{beta}$	22.1	135.6
$K_{alpha}$		

## LiF Sample

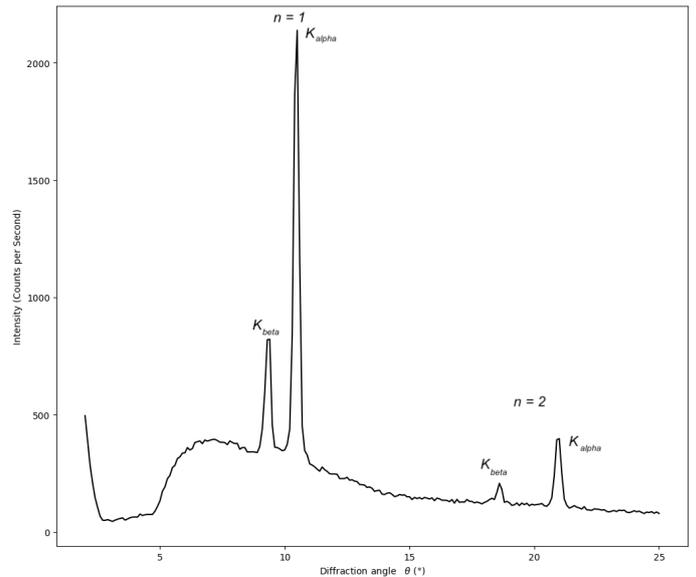
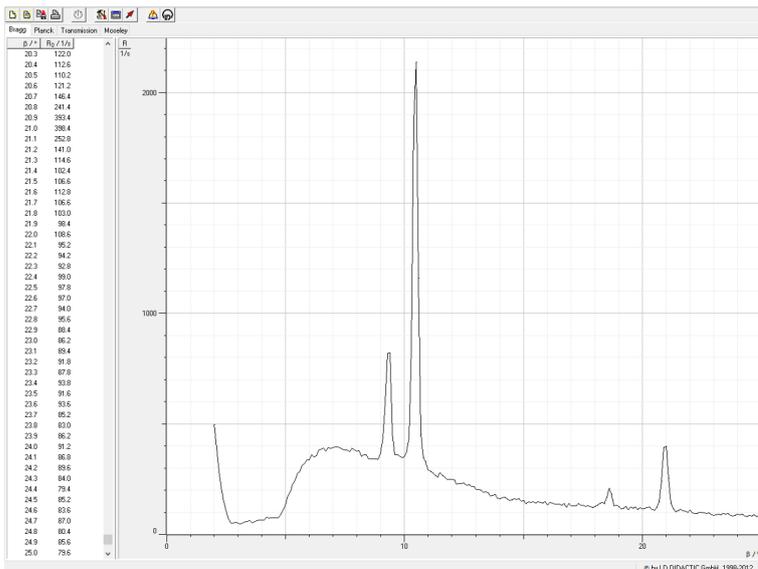


Figure 2: Graph of LiF Diffraction

## LiF Diffraction Peak Values

Values of  $n = 1$

	Diffraction Angles $\theta^\circ$	Intensity ( $R_0 \text{ s}^{-1}$ )
$K_{beta}$	9.4	821.6
$K_{alpha}$	10.5	2137.6

Values of  $n = 2$

	Diffraction Angles $\theta^\circ$	Intensity ( $R_0 \text{ s}^{-1}$ )
$K_{beta}$	18.6	207.8
$K_{alpha}$	21	398.4

### Analysis:

Theoretical value of 'd' for NaCl:

Density of NaCl is  $2.16 \times 10^3 \text{ kg/m}^3$

How many atoms in  $1\text{m}^3$  of NaCl:

$$2.16 \times 10^3 \text{ kg/m}^3 \cdot 1\text{m}^3 = 2.16 \times 10^3 \text{ kg}$$

58.5g of NaCl contains  $12.04 \times 10^{23}$

$$\frac{2.16 \times 10^3 \text{ kg}}{58.5 \times 10^{-3} \text{ kg}} = 36923.08$$

$2.16 \times 10^3 \text{ kg}$  is  $\approx 36923$  times bigger than 58.5g:

$$12.04 \times 10^{23} \cdot 36923.08 = 4.45 \times 10^{28} \text{ atoms}$$

In order to simplify this calculation we should change our distance from  $\text{m}^3$  to simply m, or in other terms change it from the number of atoms in a cube of NaCl to a single layer of NaCl. We do this by cube rooting the number of atoms:

$$\sqrt[3]{4.45 \times 10^{28} \text{ atoms}} = 3.54 \times 10^9 \text{ atoms}$$

Now if we divide the 1 meter long layer by the number of atoms in that layer, we should get a value for the distance between each one:

$$\frac{1 \text{ meter}}{3.54 \times 10^9 \text{ atoms}} = 2.82 \times 10^{-10} \text{ meters}$$

The  $\times 10^{-10}$  m suffix is common when making this type of calculation and is known as an Angstrom, represented by  $\text{\AA}$ . So  $2.82 \times 10^{-10}$  m is also known as  $2.82 \text{ \AA}$

Calculating the wavelengths of the diffracted x-rays using the Bragg equation:

<b>n</b>	$\theta \text{ K}_\alpha$	$\lambda \text{ K}_\alpha$	$\theta \text{ K}_\beta$	$\lambda \text{ K}_\beta$
1	7.3°	71.7pm	6.3°	61.9pm
2	14.4°	70.1pm	12.8°	62.5pm
3	/	/	22.1°	70.7pm

We calculated the values for  $\lambda \text{ K}_\alpha$  and  $\lambda \text{ K}_\beta$  by subbing in the angles where the peaks were located on the graph, from our 'Values of n' tables for NaCl above, into the Braggs equation (Eq.1 in method).

Calculation for n = 1:

$$\lambda = \frac{2(2.82 \times 10^{-10}m)\sin(7.3^\circ)}{1} = 71.7 \times 10^{-12}m$$

$$\lambda = \frac{2(2.82 \times 10^{-10}m)\sin(6.3^\circ)}{1} = 61.9 \times 10^{-12}m$$

Calculation for n = 2:

$$\lambda = \frac{2(2.82 \times 10^{-10}m)\sin(14.4^\circ)}{2} = 70.1 \times 10^{-12}m$$

$$\lambda = \frac{2(2.82 \times 10^{-10}m)\sin(12.8^\circ)}{2} = 62.5 \times 10^{-12}m$$

Calculation for n = 3:

[There is no data for the  $\text{K}_\alpha$  angle]

$$\lambda = \frac{2(2.82 \times 10^{-10}m)\sin(22.1^\circ)}{3} = 70.7 \times 10^{-12}m$$

We can now calculate the average value for both  $\lambda \text{ K}_\alpha$  and  $\lambda \text{ K}_\beta$

$$\frac{71.7 \times 10^{-12}m + 70.1 \times 10^{-12}m}{2} = \boxed{70.9\text{pm}}$$

$$\frac{61.9 \times 10^{-12}m + 62.5 \times 10^{-12}m + 70.7 \times 10^{-12}m}{3} = \boxed{65.0\text{pm}}$$

Comparing with given literary values:

Literary values:

	$\lambda K_\alpha$ (pm)	$\lambda K_\beta$ (pm)
Literary Values	71.08	63.09

We can use the equation to compare our values:

$$\frac{\text{Known value} - \text{Experimental value}}{\text{Known value}} \times 100 = \text{Percentage difference} \quad (2)$$

Percentage difference  $\lambda K_\alpha$ :

$$\frac{71.08 - 70.9}{71.08} \times 100 = 0.25\%$$

Percentage difference  $\lambda K_\beta$ :

$$\frac{63.09 - 65}{63.09} \times 100 = 3.03\%$$

Observation of higher order diffraction ( reflection) peaks for NaCl:

When averaging out the wavelength for  $K_\alpha$  my answer was much more accurate to the literary value than the wavelength for  $K_\beta$ . This difference is mostly due to wavelength calculated at  $n = 3$ , the graph didn't provide a third order diffraction peak for me to analyse with  $K_\alpha$  however it did give a third order peak for  $K_\beta$  which was higher than the previous two orders. By including the value at  $n = 3$  I achieved a value which was further away from the literary value.

Experimental determination of d from the first order peak of LiF:

Once again we will use the Bragg's equation (Eq. 1) manipulated:

$$d = \frac{n\lambda}{2\sin(\theta)}$$

$$d = \frac{(1)(71.08 \times 10^{-12})}{2\sin(10.5)} = 1.95 \times 10^{-10}\text{m} \text{ or } 1.95\text{\AA}$$

$$d = \frac{(1)(63.09 \times 10^{-12})}{2\sin(9.4)} = 1.93 \times 10^{-10}\text{m} \text{ or } 1.93\text{\AA}$$

Since we know the distance between the lattice spacing should be constant, we can use the average value of  $1.94\text{\AA}$  find the angle of both  $n = 2$  and  $n = 3$ .

Calculation of the higher order diffraction (reflection) peaks for LiF:

$$\theta = \sin^{-1} \left( \frac{n\lambda}{2d} \right)$$

$$K_{\alpha} \quad n = 2 : \quad \theta = \sin^{-1} \left( \frac{(2)71.08 \times 10^{-12}}{2(1.95 \times 10^{-10})} \right) = 21.49^{\circ}$$

$$K_{\alpha} \quad n = 3 : \quad \theta = \sin^{-1} \left( \frac{(3)71.08 \times 10^{-12}}{2(1.95 \times 10^{-10})} \right) = 33.34^{\circ}$$

$$K_{\beta} \quad n = 2 : \quad \theta = \sin^{-1} \left( \frac{(2)63.09 \times 10^{-12}}{2(1.95 \times 10^{-10})} \right) = 18.98^{\circ}$$

$$K_{\beta} \quad n = 3 : \quad \theta = \sin^{-1} \left( \frac{(3)63.09 \times 10^{-12}}{2(1.95 \times 10^{-10})} \right) = 29.20^{\circ}$$

The  $n = 2$  peak was visible on our LiF graph and the angles calculated here line up pretty accurately;  $K_{\alpha}\theta$   $21.49^{\circ}$  compared to the  $21^{\circ}$  we obtained from the graph;  $K_{\beta}\theta$   $18.98^{\circ}$  compared to the  $18.6^{\circ}$  we obtained from the graph.

The  $n = 3$  peak was not visible on our LiF graph, the calculation implies that in order to see the third peak on our graph we would have to increase our angle range to at least  $33.34^{\circ}$ , instead of our upper limit of  $25^{\circ}$ .

## Questions:

1. What type of bonding would you expect to find in the NaCl crystal?

We know from studying inorganic and physical chemistry that the periodic table is designed in columns and rows from left to right where the left most column has 1 electron in its outer shell and the right most column has a full 8 electrons in its outer shell. Na has 1 electron in its outer most shell and Cl has 7, when these elements bind together to form the compound NaCl instead of sharing the electron, the one valence electron is completely transferred to the Cl atom. This type of bonding is known as Ionic Bonding.

2. Explain why the angular spacing between the two x-ray lines increases as the order number increases.

As the diffracted wavelength for the two x-ray lines are different values, when you multiply both of them by the order number the difference between the two wavelengths multiplies by the same amount e.g when you take  $71.08 - 63.09$  you get  $7.99$ , when you take  $(2)(71.08) - (2)(63.09)$  you get  $15.98$ . When using the equation to find the angle, all values are equal except for wavelength so it is this difference which causes the spacing between the two x-ray lines to increase.

## Discussion:

One of the largest experimental errors associated with the apparatus would be the platform which holds the crystal sample. While the experiment was being carried out, two supervisors made reference to and adjusted how flush the crystal sample was against the platform it was placed into. Since there is no way to lock the sample in place over the course of the measurements, it is possible that slight vibrations in the machine caused the crystal sample to move and affected the diffraction pattern before or during the gathering of data.

When analysing the graph of the NaCl diffraction pattern, a slight bump in intensity was noticed before  $20^\circ$  which quite possibly could have been  $K_\beta$  meaning that the peak labelled  $K_\beta$  at  $n = 3$  could actually be alpha. I tried taking this small peak into account however both of my wavelengths decreased in accuracy to the literature value so I assumed it wasn't actually a peak. Upon reflection this post hoc analysis of my graph should probably be avoided in future as to avoid confirmation bias.

If I were to repeat this experiment with the intent of increasing my accuracy my first choice would be to run each measurement multiple times instead of just once. There was an issue with the apparatus and computer which meant time carrying out the measurements were limited and obviously having more data to work with would give a better overall representation of the diffraction patterns inherently found within NaCl and LiF. This would hopefully improve the accuracy of the results found.

## Conclusion:

Using the data provided we were able to determine the lattice constant of NaCl to be  $2.28 \text{ \AA}$ . We used this constant to find the average wavelength for both  $K_\alpha$  and  $K_\beta$  which were  $70.9 \text{ pm}$  and  $65 \text{ pm}$  respectively, these values have an error of  $0.25\%$  and  $3.03\%$  off the known literature values. We determined the lattice spacing of LiF to be  $1.94 \text{ \AA} \pm 1$  and estimated the third order peaks to be present at  $29.20^\circ$  for  $K_\beta$  and  $33.34^\circ$  for  $K_\alpha$ .