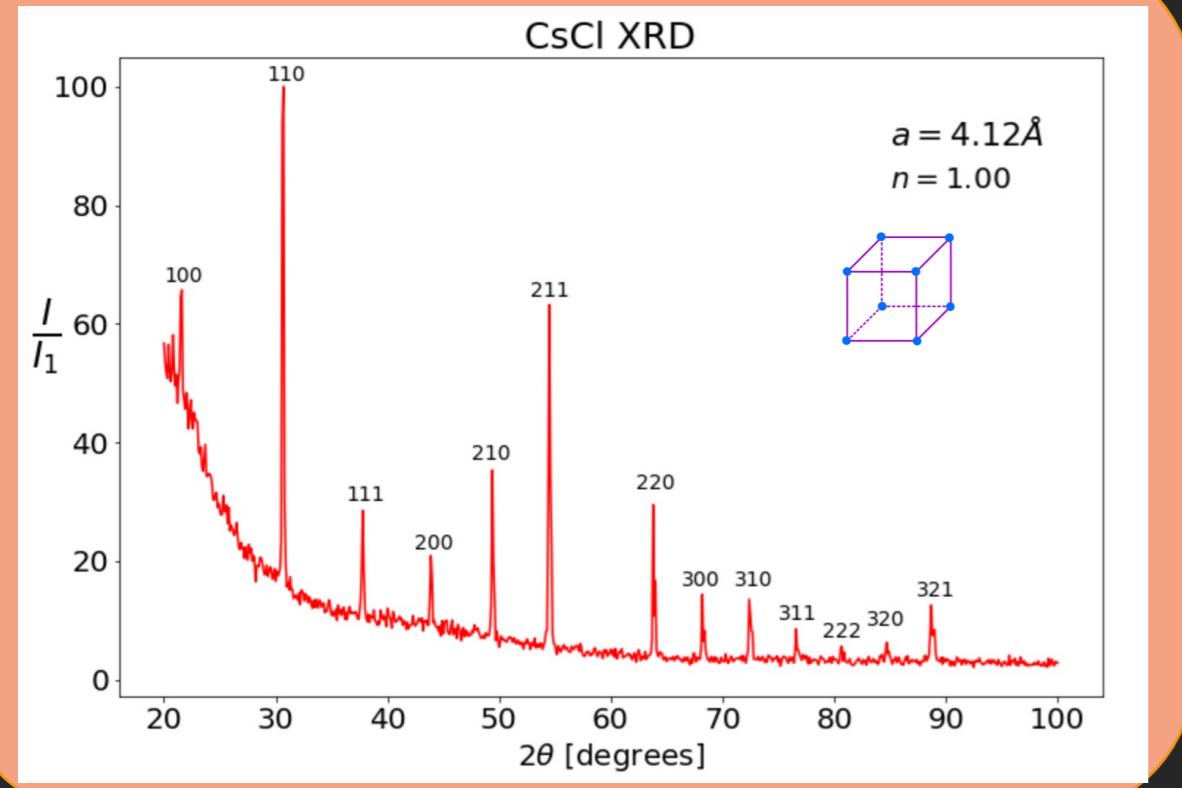
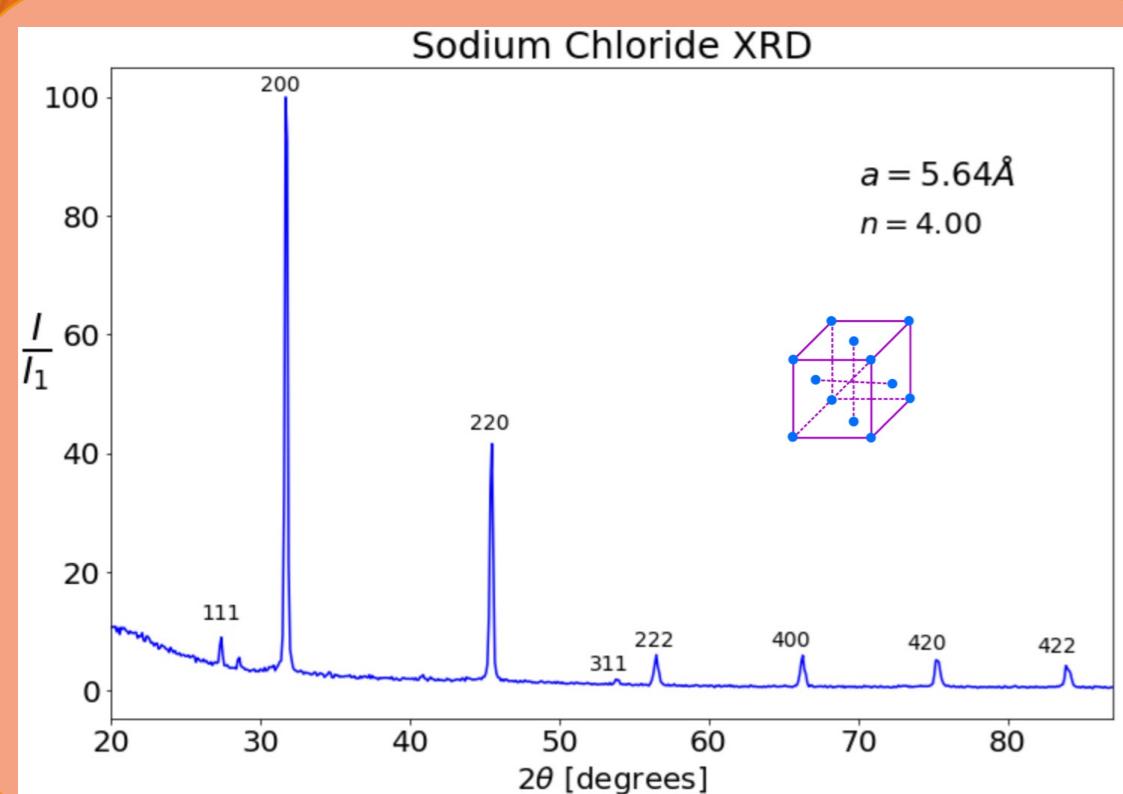
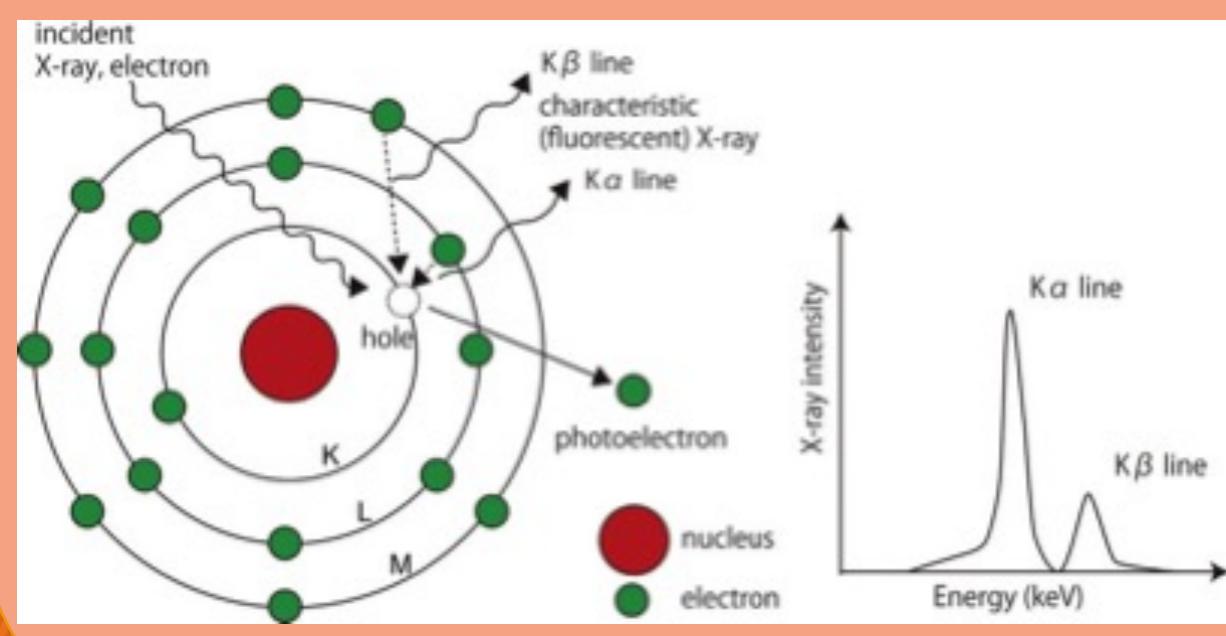


X-ray Tube

X-rays are produced in a cathode ray tube by heating a filament to produce electron via **thermionic emission**. These electrons are then accelerated towards a target by applying a potential difference.

When electrons with sufficient energy collide with an inner atomic electron, the atomic electron is ejected from the atom, leaving a vacancy. This vacancy is filled by an electron in a higher electron shell, releasing a photon equal to the energy difference of the transition as it does so. These photons are **characteristic x-rays**. Different transitions correspond to different energies, known as **K_a** and **K_b**. These x-rays



Identifying K_B peaks

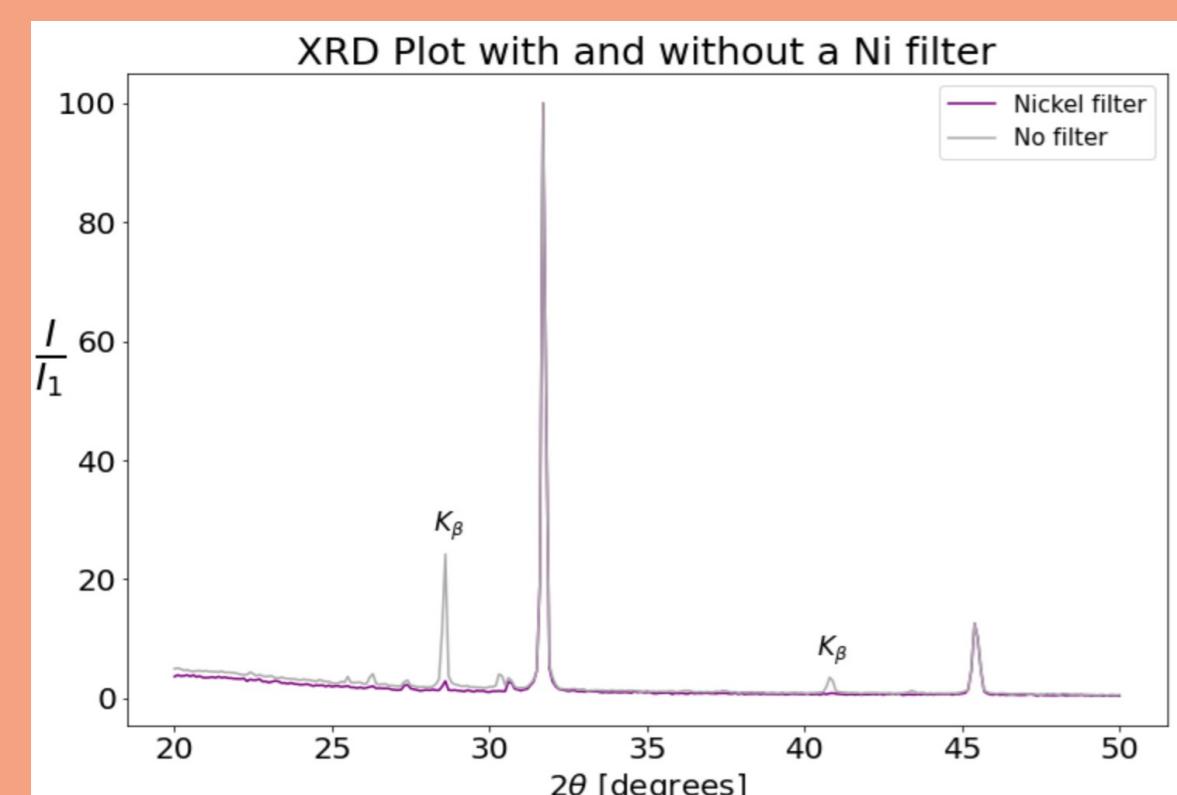
When X-rays are produced, there are three different wavelengths of characteristic x-rays that are produced:

$$\begin{aligned} Cu\ K_{\alpha 1} &= 1.54056 \text{ \AA} \\ Cu\ K_{\alpha 2} &= 1.54439 \text{ \AA} \\ Cu\ K_{\beta} &= 1.39222 \text{ \AA} \end{aligned}$$

The two K-alpha peaks are close enough that when identifying the peaks the difference is negligible.

However the K-beta waves have significantly shorter wavelengths, and so will produce a peak at a different angle, as $\lambda \propto \sin \theta$.

In our XRD's, a **Nickel filter** is used to attenuate the K-Beta waves, however, if we remove this filter then we can observe the characteristic peaks from K-Beta x-rays.

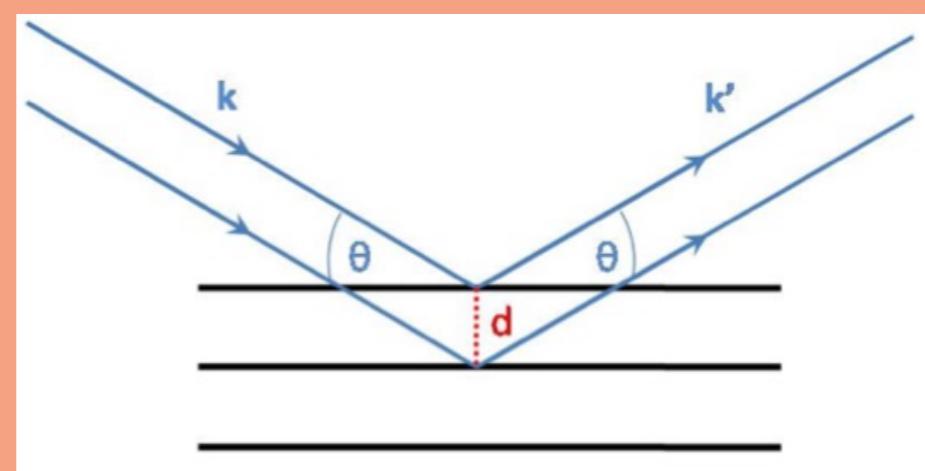


Using Bragg's Law, and the same method as before, we can determine that the peaks labelled in the graph correspond to the 111 and 220 planes. Finding these planes proves that this peak is a result of the K-Beta waves.

Bragg's Law

A crystal lattice consists of **planes**. Planes have uniform density variations separated by distance **d**. When an x-ray is incident on the plane, at some angle θ , it is reflected. If the waves from the two planes are in phase, then we get constructive interference. The condition for this is known as **Bragg's Law**.

$$2d \sin \theta = n\lambda$$



Determining lattice parameters

Using **Bragg's Law**, we can calculate the value for **d**. When **d** is known, it we can look up the values for the substance in a data book and compare it with the known value. The value of **d** should correspond to lattice parameters. When we know what the lattice parameter for one **d** value should be, we know what $h^2 + k^2 + l^2$ equals.

We then define a divisor to calculate the remaining values for $h^2 + k^2 + l^2$ and from this deduce **hkl**.

This process was used to calculate the lattice parameters for the NaCl, KCl and CsCl graphs seen to the left and above. Once these values are known, any plane can be used to determine the lattice constant **a** by simply plugging the values into the **Cubic Lattice Spacing** equation.

Cubic Lattice Spacing

For a cubic crystal, the plane spacings are given by

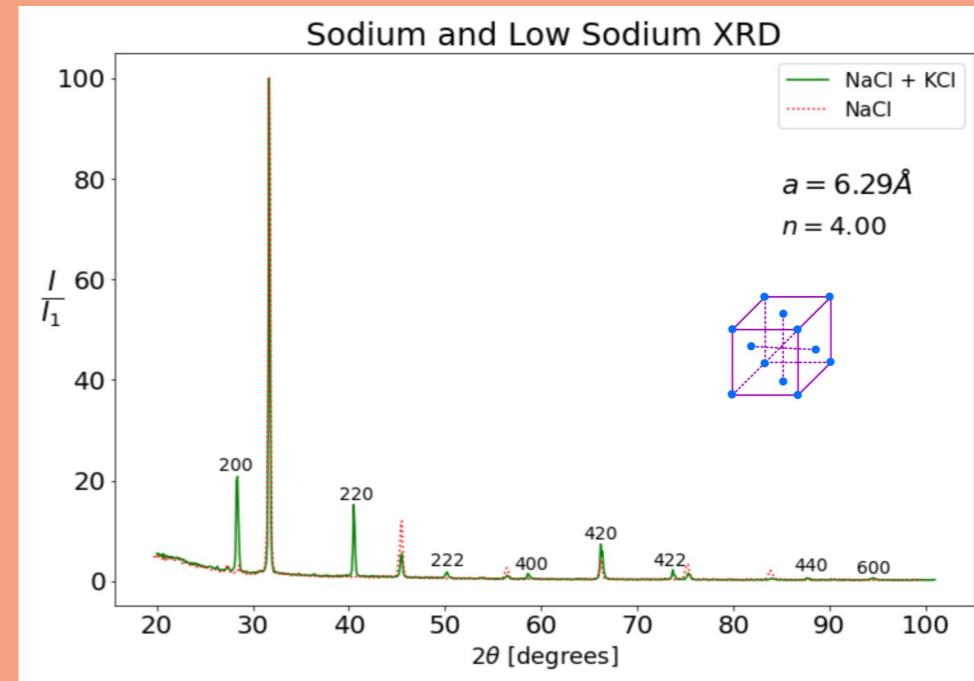
$$d = \frac{a}{\sqrt{h^2 + k^2 + l^2}}$$

Where **hkl** are lattice parameters and **a** is the lattice constant. From finding the positions of the peak, it is possible to determine the lattice parameters and lattice constant

Finding KCl

Low sodium salt contains 51% KCl. Knowing the peak positions for NaCl it is possible to find which peaks in a low sodium salt XRD belong to KCl specifically.

KCl was found to share one of the same peaks as NaCl, the 420 peak.



Güner Aygin's

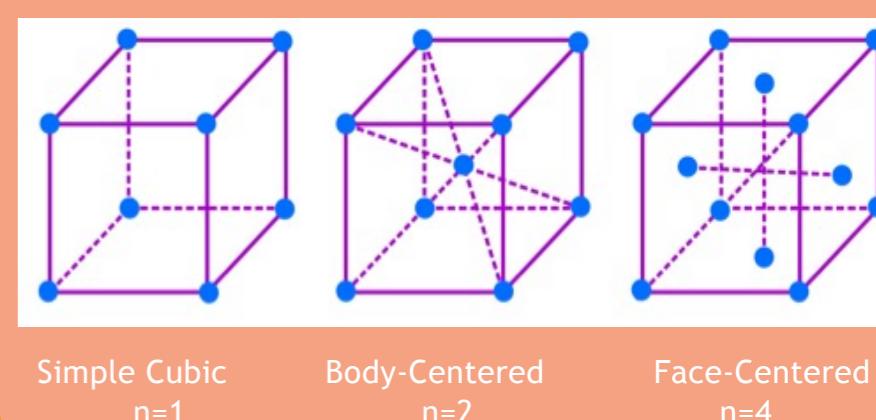
X-ray Powder Diffraction

Lattice Shapes

From determining the lattice constant, it is possible to work out the shape of the cubic structure, by calculating the number of atoms per unit cell.

$$n = \frac{N_A \rho a^3}{m}$$

Where **n** is the number of atoms per unit cell, **N_A** is Avagadro's number, **ρ** is the density and **m** is the molecular mass number. The blue dots below are **lattice points**, not atoms.



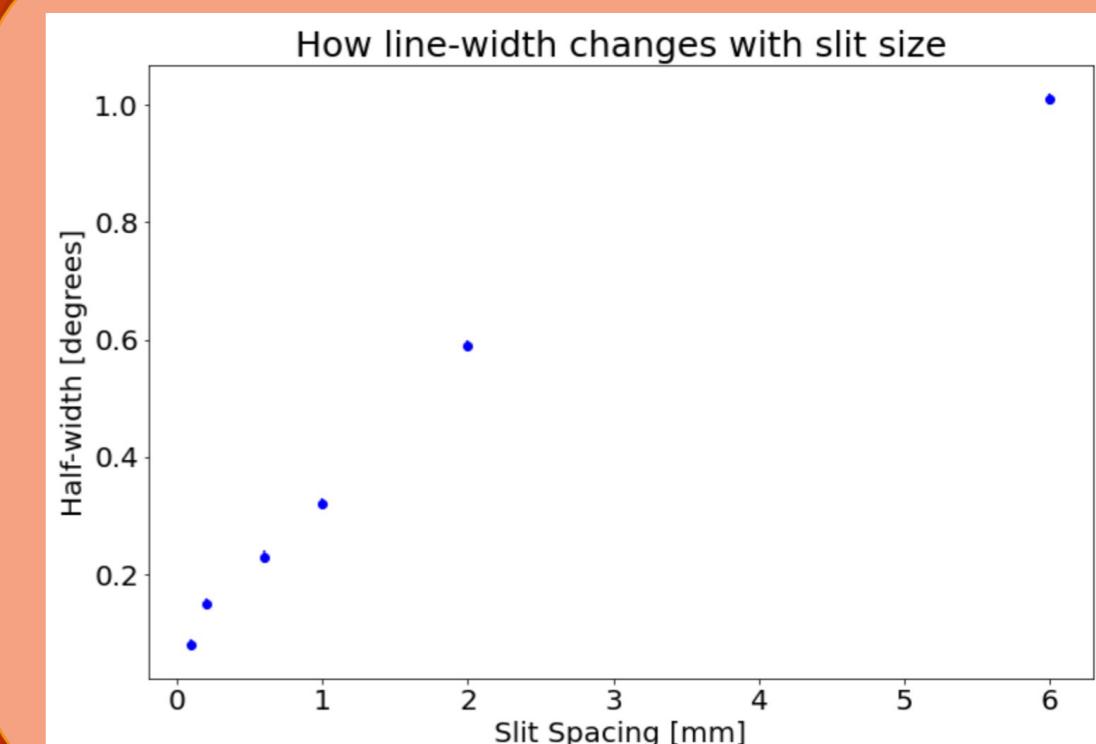
Identifying an Unknown Mineral

We can use our X-ray diffraction technique to forensically determine what substance we are studying.

In this case, we were analysing a pink mineral we believed to be **Rhodochrosite**, which is comprised of **MnCO₃**. The rock was very difficult to grind very fine, and as such the XRD was noisier than the others.

We concluded that the mineral was Rhodochrosite, but we could not use our traditional method of determining the lattice parameters. Instead, we simply had to compare the **d** values and see what **hkl** should be.

This is because, unlike the other samples we analysed, Rhodochrosite isn't a cubic, but rather is **Hexagonal**. As such, we cannot determine what the lattice constant **a** is from the cubic equation.



How changing the X-ray slits affects the XRD graph

The x-ray machine has three slits, which act as apertures. There is one slit at the source, and two at the detector: one before and one after the Ni filter.



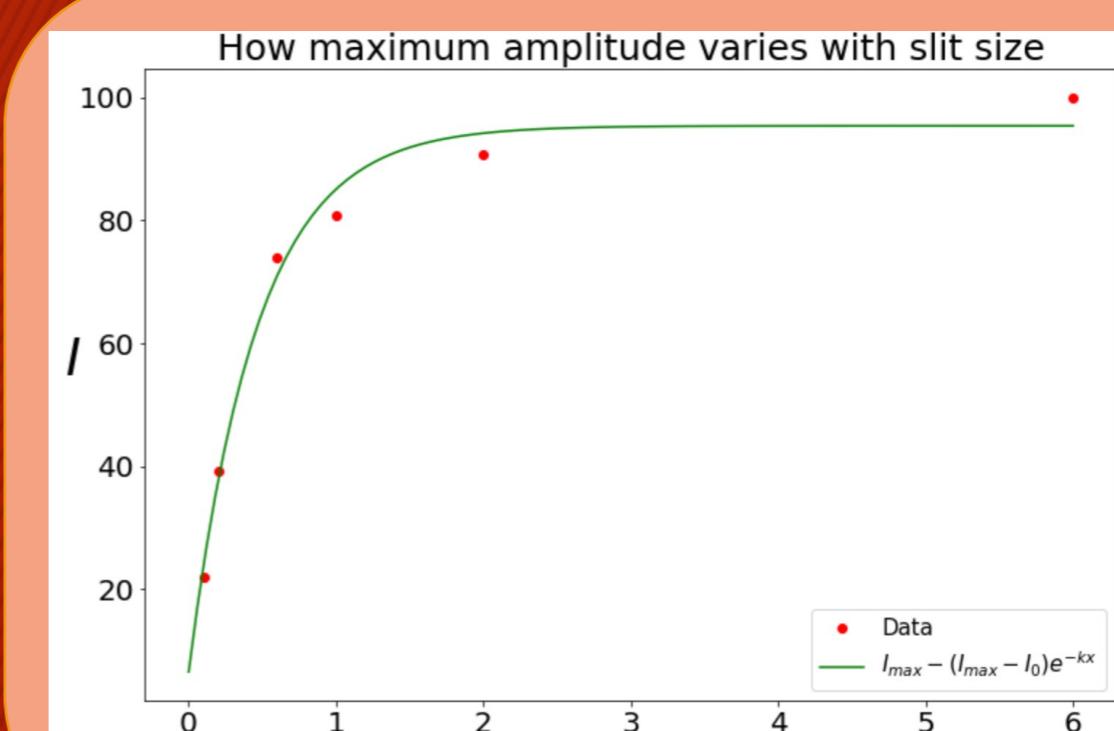
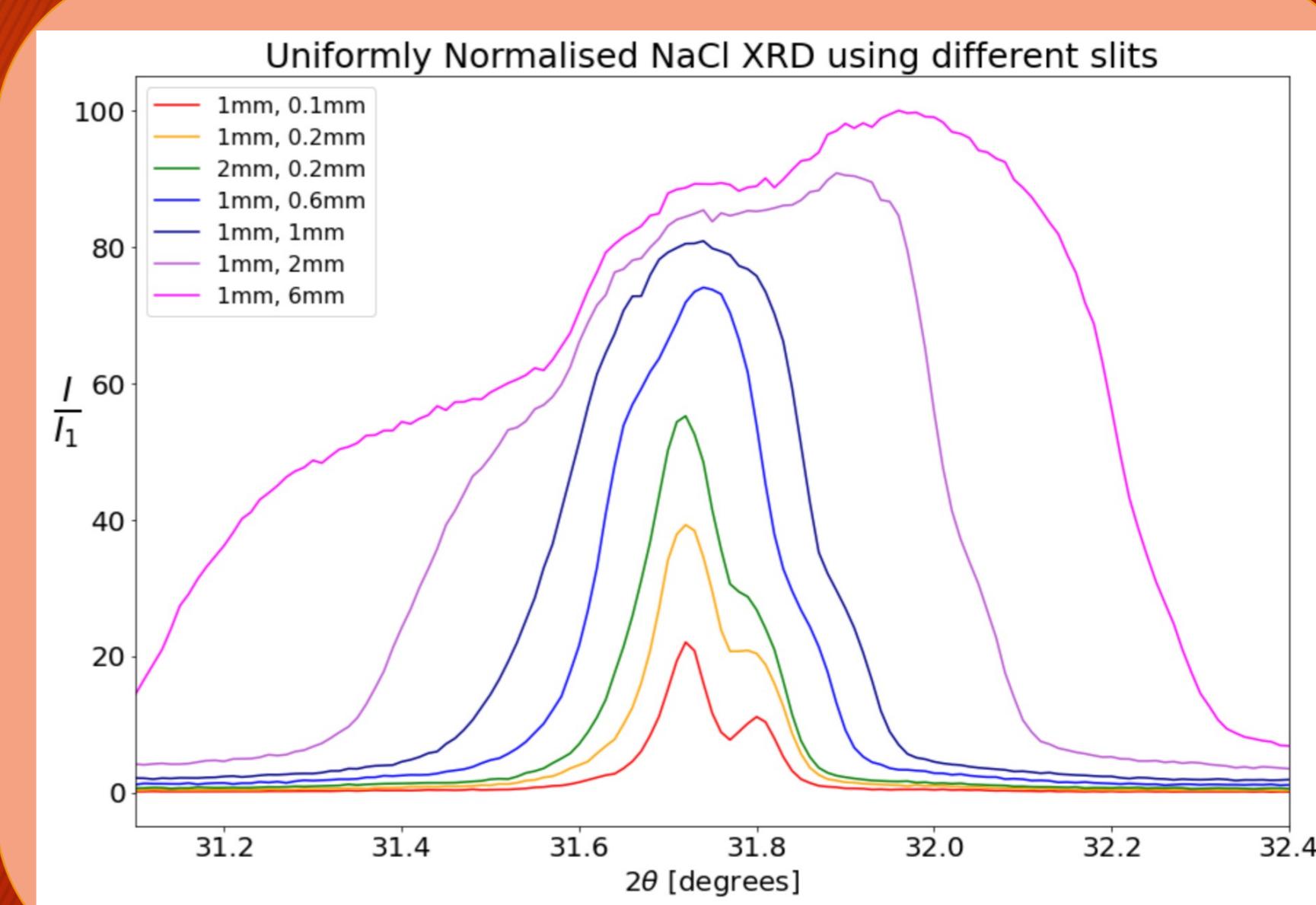
We investigated how changing the slits at the detector changes the XRD graph. In theory, we assume that as the slit spacing increases, the intensity of the signal increases, as more x-rays hit the detector. We also expect that the signal becomes noisier and loses resolution, due to the larger slit spacing. Both effects can be seen in the graph. The slit spacings are written as ('slit before filter', 'slit after filter').

We also notice that as the slit spacing increases, the line width also increases.

Too large a slit spacing significantly distorts the XRD graph. We see the alpha-1 and alpha-2 peaks in the red line, which is completely lost in the pink line, as well as the peak position.

A graph of how the maximum amplitude increases with slit size shows a plateauing exponential relationship.

A graph of how line-width changes as a function of slit spacing shows a roughly linear relationship between the two.



WARNING!

X-rays are highly ionizing and can severely burn skin and cause cancers. The x-rays in this experiment have energy of around 8keV, which are readily absorbed by bodily tissue. Significant care must be taken when working with x-rays.