

**Congratulations! The diffraction pattern from the sample that you made as part of Project M has now been collected as part of the 24 hour experiment on Beamline I11 here at Diamond!**

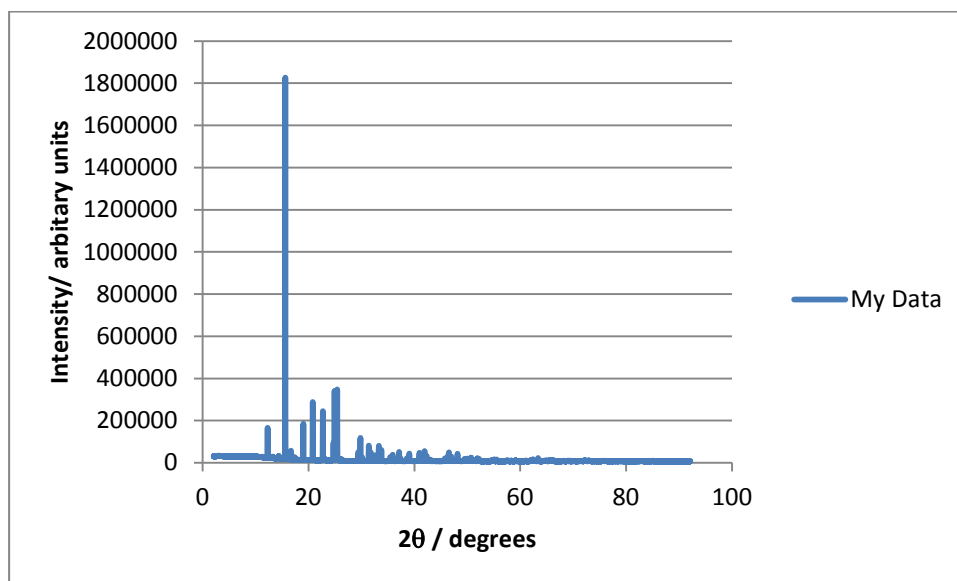
The next step, and the most important part of any experiment, is now to analyse the data from your sample. The instructions below will outline how to examine your diffraction pattern and compare to the patterns from different calcium carbonate phases (calcite and vaterite) in order to determine what phases your sample contains. You can then carry out some peak fitting to reveal more details on the crystal structure of your sample finally your sample to the rest of your school's results in order to make some conclusions about the effect of the additive you have used.

The video contains further instructions on how to login to the Project M webpage, plot the diffraction patterns from your sample and the standards, fit peaks and enter your results.

You can also download your data file using the Download button. The data files as collected during the experiment are .dat files. The data inside the file is a three column dataset for your x value (two theta in degrees), your y value (intensity) and the error in the intensity. To open this data in your preferred software package, download the file and rename the extension from .dat to .xye. This will enable the data to be imported into most plotting programs including Microsoft Excel, Open Office etc.

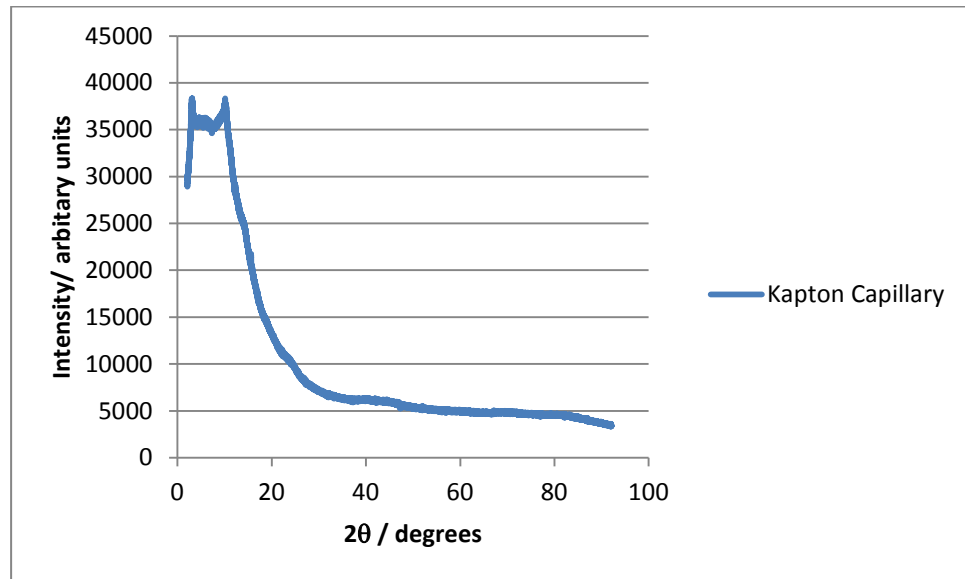
#### 1. Looking at your data

You will have a plot of diffraction angle ( $2\theta$ , sometimes written as 2 theta), on the x axis vs intensity, on the y axis and should see a pattern that looks something like this:

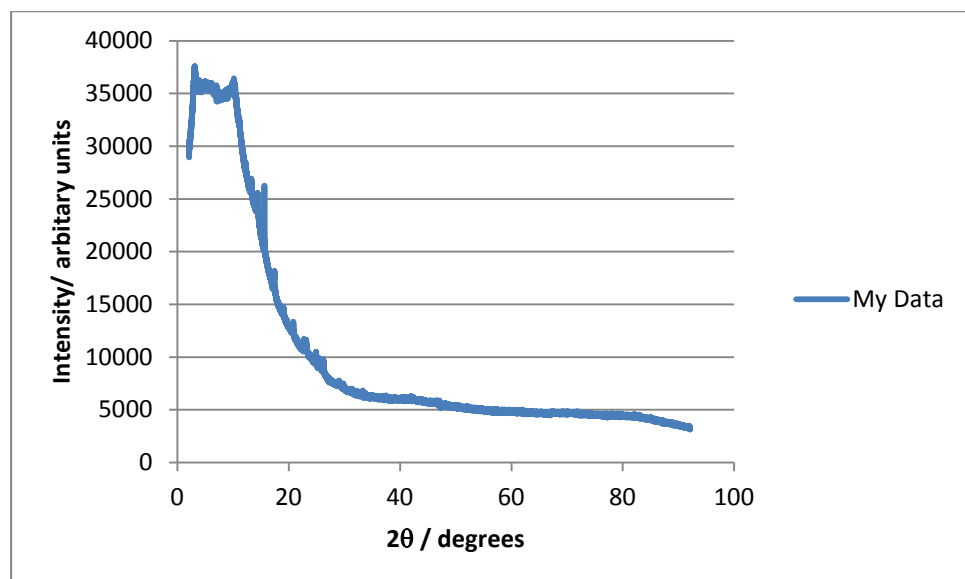


The kapton capillary tube that you loaded your sample into contributes to the diffraction pattern. A pattern of an empty tube is shown below, it is essentially some

broad bumps in the background (note the scale on the y-axis compared to the sample pattern).

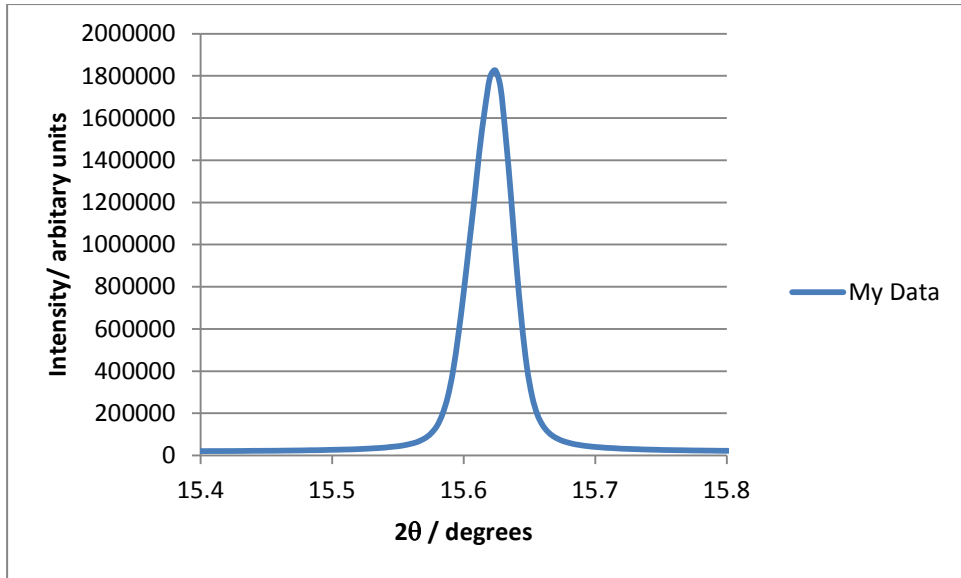


For most samples the peaks should appear much larger than this background, however if the sample was loosely packed or there were some gaps in the tube there may not have been enough crystals in the x-ray beam to give the strong diffraction peaks, so you pattern may look more like this:

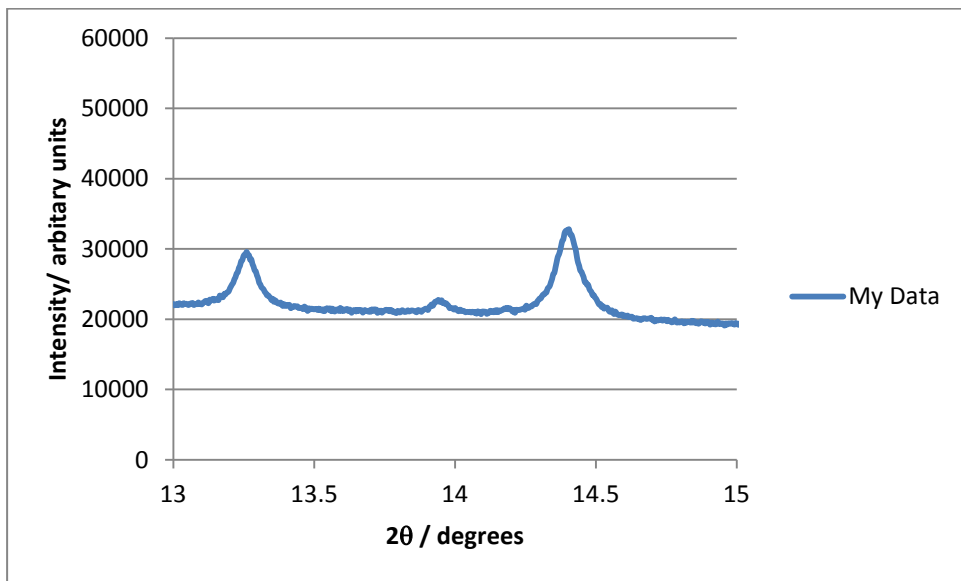


Don't worry! You should still be able to use the pattern to work out what your sample is!

Zoom in on a few of the peaks, try the largest (most intense) peak first. What does it look like? You should see a smooth peak outline, rather than a jagged line (a smooth hill, not bumpy mountain peaks!).

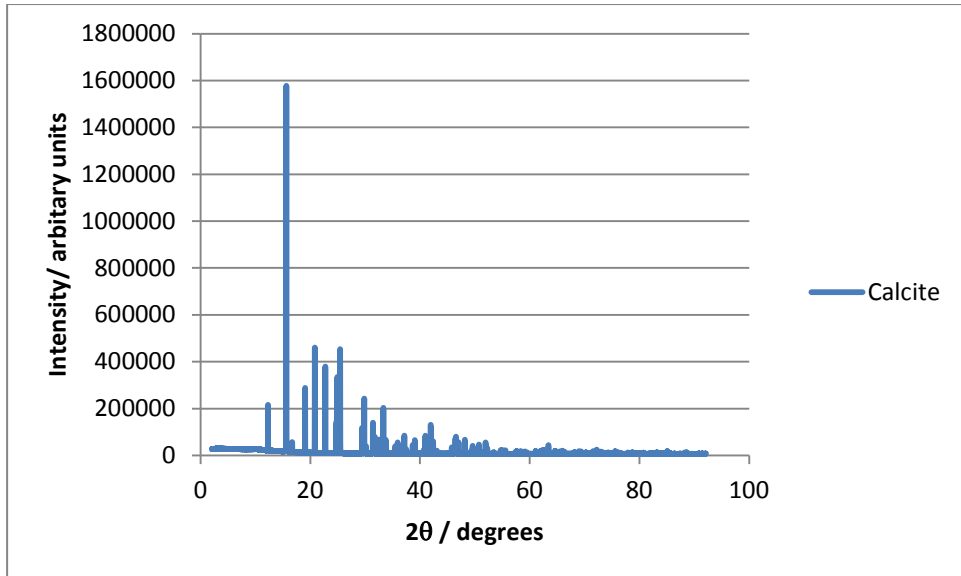


Zoom in on the background and check for any less intense peaks.



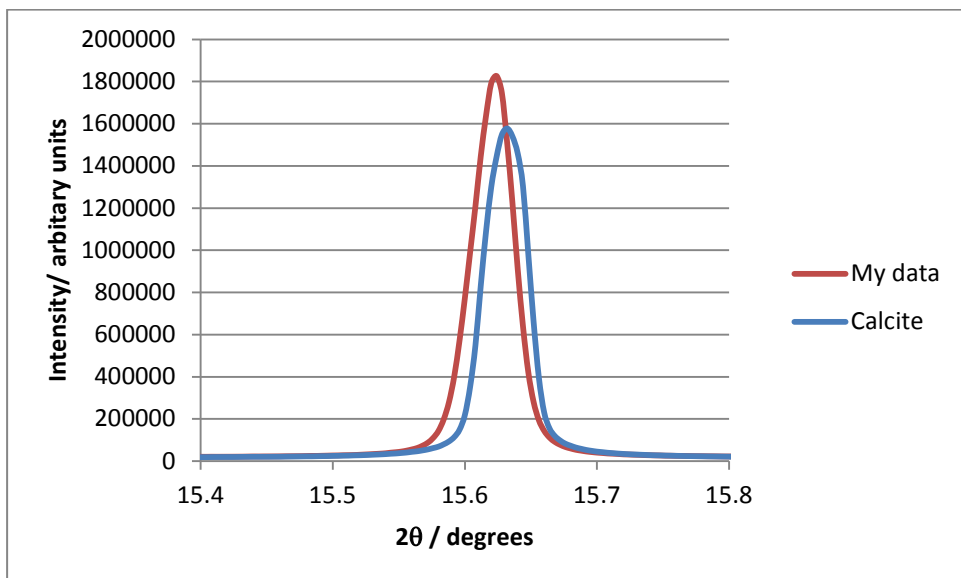
2. Have you made calcite or vaterite (or both!)?

Plot the calcite diffraction pattern. Make a note of the 2theta positions and intensities of the 3 strongest peaks.



Now compare to your diffraction pattern, do you have these peaks? If so then you have calcite in your sample!

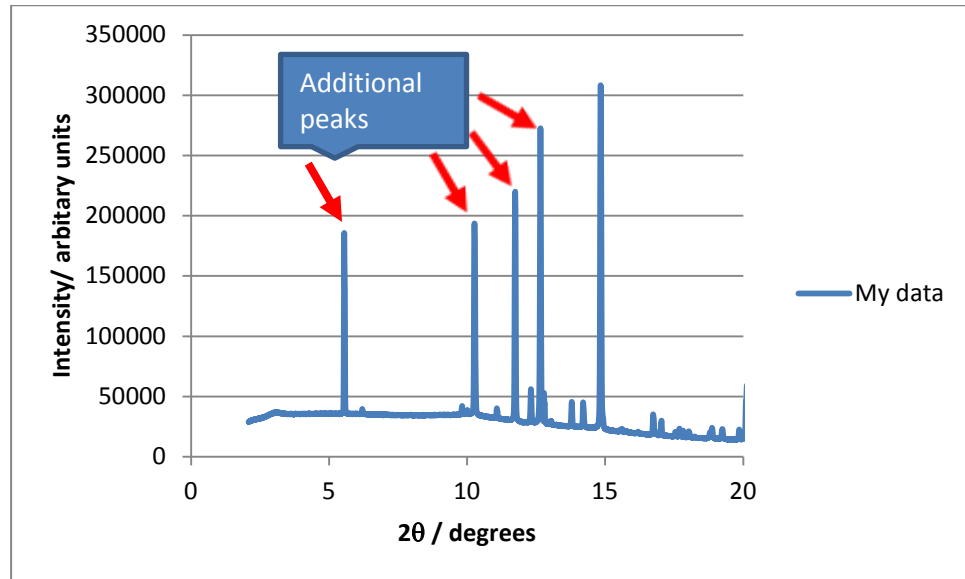
**IMPORTANT:** The intensities in your sample will NOT be the same as those in the standard, as you will not have the same amount of calcite in your sample, however the intensity RATIO should be similar. For example, if peak 2 is half the intensity of peak 1 in the standard then it should be half the intensity in your sample. Don't worry if the peak position is not exactly the same but it should be close (within about 0.2 degrees)



Repeat for the vaterite standard. Do you have vaterite in your sample?

If you have both calcite and vaterite divide the intensity of your calcite peak by the intensity of the vaterite peak to calculate a calcite/vaterite "ratio" for your sample.

Are there any extra peaks in your diffraction pattern that aren't vaterite or calcite?



What do you think they could be? How could you check this?

3. The exact peak position of the vaterite and calcite diffraction peaks is determined by the layer distances within the crystal structure. These may change if the additive becomes included in the crystal structure.

Use the peak fitting tool to fit the calcite peak at 15.6 deg, update the peak position field with this value.

Repeat for the vaterite peak at 14.4 deg

Are your peaks at lower or higher angle than those in the standards?

4. Comparing to other samples.

How does your sample compare to others that were made with the same amount/type of additive? Plot these on the same plot as yours and compare the peaks positions and relative intensities. Note any similarities and differences. Why do you think they might be different?

The samples made by your school should all have been made with the same additive, in increasing concentration (apart from the tap water sample). Compare your sample to the others from your school using the plot, and share the information you have already calculated about the peak positions and calcite/vaterite peak ratio with the rest of the class.

Are there any trends on increasing the amount of additive?

How is the tap water sample different from the sample made with just distilled/deionised water?