X-ray diffraction:
Determining lattice constants of crystal structure

Purpose:

This experiment will show you how to perform an x-ray diffraction experiment in order to measure the structure of a crystal. In particular you will do a Bragg diffraction experiment on two different crystals in order to measure the lattice constants of the structure, detecting the K_α and K_β peaks.

Introduction:

You will use the Leybold x-ray spectrometer in the Bragg diffraction mode to study the crystal structure of two different crystals: sodium chloride (NaCl) and potassium bromide (KBr), both cubic crystals. The basic theory involves the Bragg condition:
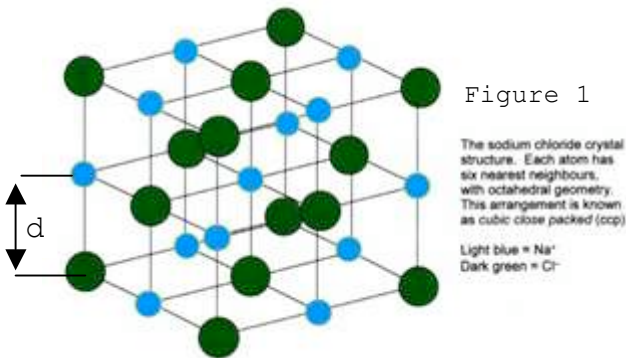
$$n\,\lambda = 2\,d\,\sin\theta, \tag{1}$$

where n is the order, λ is the wavelength, d is the lattice plane spacing, and θ is the diffraction angle. Note that the lattice plane spacing corresponds to half of the unit cell dimension for a cubic crystal as seen in Figure 1. Thus we have that

$$d = a_o/2, \tag{2}$$

where a_o is the lattice constant of the cubic crystal. Given this, we can re-write Equation (1) to have a working equation for this experiment:

$$n\,\lambda = a_o\,\sin\theta. \tag{3}$$

Figure 1

The sodium chloride crystal structure. Each atom has six nearest neighbours. with octahedral geometry. This arrangement is known as cubic close packed (ccp).

Light blue = Na+
Dark green = Cl-

So if we are able to obtain the diffraction angles for known x-ray wavelength peaks of various orders, we can calculate the lattice constant for our crystal.

Pre-Lab Exercises:

1) Look up and write a paragraph about how an x-ray tube functions. What determines the x-ray wavelengths?

2) Derive Equation (1) by considering interference from "reflections" from two neighboring crystalline planes in a crystal.

3) Look up the lattice constants for your two samples and make a table of the expected diffraction angles for the first 3 orders for the K_α and K_β x-ray peaks for each sample using Equation (3).

These should be written up separately and handed in at the laboratory session when the experiment is done.

Procedures:

Overview of experiment:

A schematic of the experiment is shown in Figure 2. The x-ray tube has a molybdenum anode so that the emitted x-rays have the following characteristic wavelengths: $K_\alpha = 71.08$ pm and $K_\beta = 63.09$ pm. These x-rays are collimated into a beam by the collimating slit and the incident beam makes an angle θ with the surface of the crystal sample. The detector, a Geiger-Müller tube is mounted in such a way that when the sample is rotated, the detector always makes an angle of 2θ with respect to the incident beam. This geometry insures that the Bragg condition will be met – namely the incident and "reflected" rays make equal angles from the crystal planes. The crystal is rotated in discrete angular steps, with the detector synchronously rotated to maintain this condition, and data are recorded at each angle for some fixed, assigned duration. The operator sets the angular step and the minimum and maximum angle to be measured and the instrument steps through these angles recording and storing the results and displaying them on a graph in the software package on the interfaced computer.
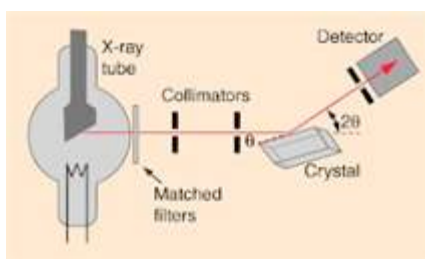

Figure 2. Schematic of experimental set-up.

Experimental Details:

Turn the x-ray apparatus main power switch on and bring up the software package on the computer. Mount the crystal sample (start with the NaCl sample), being careful to only hold it by the side edges. Note that these crystals are hygroscopic (absorb water) and extremely fragile. They are stored in a dessicator and should be handed very carefully. Place the crystal on the target stage, carefully raise the platform all the way to the stop and tighten the knurled screw. Close the lead glass protective doors. Set the tube voltage to 35 kV and the emission current to 1.0 mA. Set the angular step ($0.1°$), minimum ($2.5°$) and maximum angles to be scanned, and the measuring time per step, Δt. Start the measurement by pressing the SCAN key. The data are plotted as received in the software graph. When the scan is completed, you should save the measurements in a desktop folder that you create. To locate the peaks, right click the mouse to select "Calculate Peak Center". Using the left mouse button, mark the "full width" of each peak and write down the center values and their SD, shown in the lower left. Make a table of your results that will enable you to make a plot of Equation (3) in order to find a best value for the lattice constant with its uncertainty. Repeat the measurements for the KBr crystal sample. [Suggestion: The diffraction is weaker for this sample and it may help to identify the peaks if you make predictions of the diffraction angles from the known lattice constant.]