# X-ray Diffraction - Additional Experiment

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## Identification of a simple rock-salt

You will now measure a rock-salt structure and attempt to determine the elements. We will provide you with three single crystals of this substance. While each sample has identical chemical and crystal structure, and appears visually as identical thin wafers, the "cut" of each crystal is made along a different axis. This is a basic rock-salt material with the same structure as table salt (NaCl). Rock-salt structure essentially is two "nested" and offset face-centered-cubic structures. For NaCl it means a Na fcc unit cell and then also a Cl fcc unit cell that is offset in one direction by $a/2$. This is shown in Figure 1.

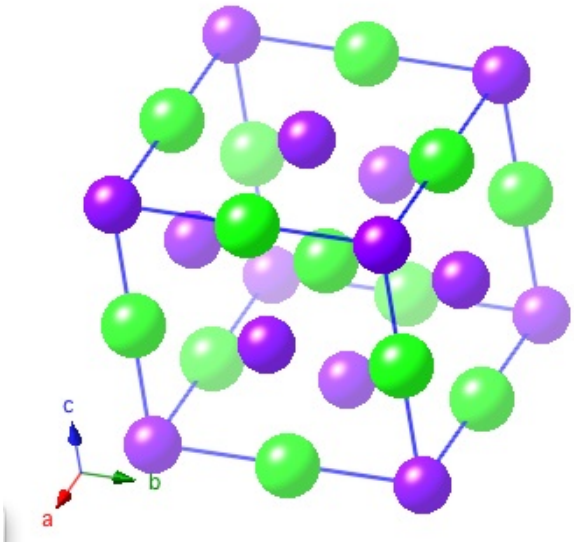


Figure 1: The rock-salt structure. Your mystery salt has the same basic structure.

You should measure the each mystery sample over a range of 10° to 50° for $\theta$ (20° to 100° for $2\theta$). You will use the same power settings on the instrument as before, and also be certain to use the Ni-filter.

Because each of the three samples is a single crystal, very few diffraction peaks will show

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up in any given scan. However, the peaks should be clearly visible when they do appear. You should be able to collect data relatively quickly at a rate of 2 sec / point. Because each is a single crystal, and each has a different facet for the large square surface, you will need to combine the scans before attempting to index the substance. You will also need to be cautious about the Al sample holder making some small spurious peaks. The small spurious signals from the Al might be seen at $2\theta$ of 38.5, 44.7, or 65.1degrees (though often they will not appear at all).

Once you have all the data, you should determine the allowed peaks for a rock salt structure.

First, recall that the Intensity is proportional to the square of the scattering amplitude. The structure factor F is a part of that scattering amplitude, hence:

$$I \propto |F|^2 \tag{1}$$

and from the appendix, the structure factor F is defined as:

$$F = \sum_n f_n e^{2\pi i(u_n h + v_n k + w_n l)} \tag{2}$$

where $h$, $k$, and $l$ are the Miller indices of the particular diffraction condition and $u$, $v$, and $w$ are the fractional positions within the unit cell of each atom. This equation is actually what gives rise to the columns for the allowed diffraction peaks in the appendix material that you used earlier.

We can further approximate this (in this case) using $Z_n$, the atomic number of that atom, in place of $f_n$, the atomic scattering factor. This is not always a valid approximation to make but it is fine here. For a simple fcc material then the structure factor is:

$$F_{fcc} = Z(1 + e^{i\pi(h+k)} + e^{i\pi(k+l)} + e^{i\pi(l+h)}) \tag{3}$$

What is $F$ for a rock-salt? Use this for integer values of (h,k,l) to determine the allowed diffraction peaks.

At this point you should be able to determine what the lattice plane each of the individual crystals possesses normal to the large square facet.

Using this information you should be able to calculate a lattice parameter for the substance and make a reasonably educated guess as to the chemical composition of the samples. If you want a little help, consider that the samples are both relatively safe to handle and also are kept with a desiccant to reduce the effects of water exposure. The material is a fairly common form of rock-salt, but not the most common.