Solid State Physics: Experiment #3
X-ray Diffraction - Single Crystal Bragg Method

This experiment uses the Tel-X-Ometer (580) X-ray instrument located in Colton 14.
You MUST sign and return to me the X-ray information handout before using this instrument!

General instructions and information about the Tel-X-Ometer are found in the Teltron manual, entitled "The production, properties, and uses of X-rays", which is located near the instrument and is to remain in Colton 14. Various parts of the present experiment are described in Sections D14, D22, and D25 of the manual. The goals for this experiment are to determine the lattice constants for four different single crystals from Bragg scattering data off a primary cleavage plane (i.e., the {100} planes} and then to locate and index scattering peaks from several {*nm*0} lattice planes. The scattering will be due to both the Cu K_α and K_β radiation, although we can distinguish these with the appropriate use of filters. To detect the scattered radiation we will use a Geiger-Muller tube. **As always, be sure to keep detailed records of your lab work in your lab notebook.** I will be checking these notebooks through the term.

For Operating the Tel-X-Ometer: See the instructions for experiment #2 (which are posted in Colton 14) and see Sections 6.5-6.6 on pg. 2 of the manual. In this experiment we will be running the X-ray tube at 30 keV.

The X-Ray tube current should be monitored at all times with a digital multi-meter.

The X-Ray tube current should not be allowed to exceed 80 μA.

For Operating the Geiger-Muller tube: See sections 10.10 and 11.0 (pg. 5) of the manual. The voltage supplied to the GM tube is monitored on channel #3 of the alarmed meter (scale reads from 0– 500V). The tube should be maintained above the threshold voltage of 370V. I suggest running at about 390 or 400V. [If you get weird readings on the alarmed meter, wiggle it around to reseat the connections with the base unit]. Channel #1 of the alarmed meter gives you the counts per second from the GM tube. (A negative reading indicates (?) < 1 cps).

Experimental Procedure:

Part 1. I(θ) vs. 2θ for {100} scattering

Here we are essentially following section D14 in the manual (pg. 17). The X-ray tube should be operated at about 50μA.

Setup: The basic experimental setup is as follows:

1 mm slot primary beam collimator (582.001) in vertical orientation
3 mm slide collimator (562.016) in slot 13 of movable carriage arm
1 mm slide collimator (562.015) in slot 18 of movable carriage arm
GM tube in holder in slot 26 of carriage arm (tube should abut 1mm slide collimator) oriented so cable in on top (tube window in vertical orientation)

Alignment: Before mounting a crystal in the crystal holder, check that the slave plate is properly zeroed. To do this, open the Tel-X-Ometer shield and move the carriage arm to the $2\theta=0$ position. The etched line on the slave plate should be aligned with the 0 on the small θ-scale (see the Fig. 1 on pg. 0 of the manual). Actually, our instrument is a bit mis-calibrated and for proper zeroing, the slave plate should be at about 1 or 1.5º (clockwise rotation). To change the slave plate angle, loosen the knurled clutch plate (again, see Fig. 1) and rotate the crystal mount while holding the carriage arm in the $2\theta=0$ position. Then retighten the clutch plate.

Crystal Mounting: See pg. 6 of the manual for details on mounting the crystals. We will first study the NaCl crystal since there is data in the manual we can compare with (see Fig. D14.7). Mount the large NaCl crystal with yellow side up and ground (not clear) main face towards X-ray tube. Note: this crystal is broken and held together with a piece of tape, so handle with care ... Also, do NOT touch the large faces of the crystals with your fingers.

Data Taking: See D14.7. Move the carriage arm through its full range taking sufficient data to create a plot like that shown in the manual. Before taking a full data set make sure you get at least 400 cps on the large scattering peak around $2\theta=30º$. If your maximum count rate is below 400 cps try remounting the crystal. If that doesn't help, you probably need to tweak the slave plate angle as described above. As you take the data, be sure to locate the peaks as accurately as possible and note down estimates for the uncertainty in the count rate.

Your NaCl data set contains Bragg peaks due to both Cu K_β and K_α radiation. We can use a nickel filter, which absorbs K_β but transmits K_α radiation, to discriminate these. Insert the Ni filter (564.004) into slot 17 of the carriage arm and take a new data set for NaCl. Again take sufficient data to make a complete plot of $I(\theta)$ vs 2θ.

We will now take data on our other three large single crystals (LiF, KCl, RbCl) as described in Section D22 (pg. 26) of the manual. For these we are just interested in locating the diffraction peaks as accurately as possible, so full data sets are not required. Take the data without the Ni filter and, for KCl and RbCl, crank the X-ray tube current up to 80μA.

Data Analysis: Using all diffraction peaks from both the K_β and K_α radiation compute the lattice spacing d for each of the four crystals. The conventional unit cell for these crystals is that of an **fcc lattice** with side length $a=2d$ (see Fig, 2.13a in Myers or Kittel Fig. 1.17). Determine the Miller indices (n00) associated with all your observed reflections.

Part 2. Scattering from (nm0) planes

Here we are essentially following section D25 in the manual (pg. 28-30). The X-ray tube should be operated at 80μA. Same setup as above. Intensities may be low so work without the Ni filter.

First, just to get further verification that we're working with cubic crystals (note that we have cleavage information for NaCl and our Laue data for LiF which both suggest cubic symmetry) we will obtain scattering data from the {010} type planes. To obtain this data we simply mount our single crystals "sideways" (i.e., rotated by 90º in the crystal mount) as shown in the sketch of D25.1. In this configuration the skinny face of the crystal is towards the X-ray beam. Locate the {010} diffraction peaks for each of the four single crystals.

Now the tricky part. Here we'll try to find scattering from some other (*nm*0) planes for the NaCl crystal. To do this we must rotate the crystal mount (i.e., the slave plate) to allow for the correct scattering geometry (like problem A2, part b on P.S. #3). For scattering off the (*nm*0) plane we must rotate the slave plate by an angle θ which is equal to the angle between the (100) and (*nm*0) planes. (In Section D.25 of the manual this angle is referred to as angle SPQ). Before attempting to take data, create a table like that shown in section D25.9 for NaCl. In this table angle SPQ=θ is the slave plate angle and 2θ is the expected scattering angle for reflections from the (*hk*0) plane. Now that you know what to look for, try to find it! A few important points. When you rotate the slave plate remember that the "zero" position is NOT at θ=0 (recall it's more like θ=1 or 1.5º). Also, to get reasonable data you need to mount the crystal in an offset position as shown in D25.10 (however, I think the manual description is backwards here ... have the corner nearest the GM tube about 1mm from the center notch on the crystal mount post). I found it very difficult to get reflections from the (*nn*0) planes but, with some work, I could find reflections from other planes (although with peak count rates around 30 cps). If you can't find any peaks, try tweaking the slave plate angle ... the whole setup is extremely sensitive to the proper geometry.

Finally, you may also want to try for some (*nm*0) peaks from the LiF crystal.

The Write-up:

You should include a discussion of how the GM detector works (see an intro physics book) and a clear drawing of the scattering setup. Your results will include I(θ) vs. 2θ plots of NaCl with and without the Ni filter, a table of scattering peaks for the other three crystals, calculated lattice constants (with uncertainty estimates) for all four crystals, and Miller indices for all Part 1 scattering peaks (constructed with reference to the conventional unit cell with side length $a=2d$). For part 2, give a table of all (*nm*0) reflections observed (include the peak GM count rate for each of these). Include your prediction table (D25.9) and note which peaks were observed and which were not. Explain why many of the planes listed in this table do not give scattering.

Write-up due date: Friday, Feb. 28.