Solid State Physics
X-ray Scattering I: Single Crystal Bragg Method

This experiment uses the Tel-X-Ometer (580) X-ray instrument located in Colton 14.
You MUST read, sign, and return 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} family of planes) and then to locate and index scattering peaks from several (*hk*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.**

Operating the Tel-X-Ometer (see Sections 6.5-6.6 on pg. 2 of the manual):

1) Turn the power on key 90° to the right. For power to be on, the timer must also be on. When the power is on, the white POWER ON lamp will be lit. In the power-on mode, current is supplied to the filament in the X-ray tube, however, no X-rays are being produced. Allow the X-ray tube to "warm-up" for at least 10 minutes. (If a crackling noise is heard when the X-rays are turned on, the tube probably needs more warming up).

2) Once the filament is ready, be sure the large plastic shield is closed and in the center locked position. To turn on the X-rays, push the red X-RAYS ON button (this supplies high voltage to the X-ray tube). If the X-RAYS ON lamp does not light up, check that the shield is in the locked position and try again.

The X-ray tube can be run at two different voltages 20 or 30 keV. There is a switch on the top of the instrument under the large plastic shield to set this. We will be working at 30 keV.

3) Once the X-rays are on, the intensity of the beam can be varied by adjusting the X-ray tube current via the inset screw to the left of the X-RAY ON button. In this experiment we will be working with a current between 50 and 80 μA.

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. (You may have to move the decimal point to the right).

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 near 60 μA.

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 very slightly mis-calibrated and for proper "zeroing", the slave plate needs to be set between 0.5-1.0° (counterclockwise rotation). To change the slave plate angle, loosen the knurled clutch plate (again, see Fig. 1) and rotate the slave plate (pushing with your thumbs) 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 (yellow) 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 (100)-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 $2\theta \approx 30°$. If your maximum count rate is below 400 cps try remounting the crystal with the other (100)-face towards the X-ray tube. If that doesn't help, you probably need to tweak the slave plate angle as described above. (You should be able to get a peak count rate of near 500 cps). 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_α and K_β 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(θ) vs 2θ.

We will now take data on our other three large single crystals (LiF [blue], KCl [green], RbCl [red]) as described in Section D22 (pg. 26) of the manual. For these we are mainly interested in locating the diffraction peaks as accurately as possible (with uncertainty), however, you should take enough data to be able to plot I(θ). Take the data both with and without the Ni filter for LiF. For the KCl and RbCl don't use the filter and nudge the X-ray tube current up to 80 μA. For each of these crystals see which (100)-face gives the larger count rate and use that face.

Data Analysis: Using all diffraction peaks from both the K_α and K_β radiation compute the inter-atomic spacing d for each of the four crystals via the simple Bragg relation $n\lambda=2d\sin\theta$ (where n is the "order" of the diffraction peak). In X-ray structural analysis it is usual to treat the crystal unit cell, rather than individual atoms, as the basic scattering elements and to describe nth-order diffraction from the (100)-planes as 1st-order diffraction from the (n00)-planes. The conventional unit cell for the alkali-halide crystals is that of an **fcc lattice** with side length $a=2d$ (see NaCl model in Colton 14; white/red beads show fcc ordering). Determine the Miller indices (h00) associated with all your observed {100} reflections using the fact that the "Bragg" condition for scattering from the (hkl)-planes is

$$\lambda = 2d_{hkl}\sin\theta \tag{1}$$

where, for a cubic crystal, $d_{hkl} = a/\sqrt{h^2+k^2+l^2}$ is the inter-plane distance.

Part 2. Scattering from (hk0) planes

Here we are essentially following section D25 in the manual (pg. 28-30). The X-ray tube should be operated at 80 μA. 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 the shape of our large single crystals strongly suggests cubic symmetry) we will obtain scattering data from the {010} family of 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 (010)-face of the crystal is towards the X-ray beam. Locate the {010} type diffraction peaks for each of the four single crystals and calculate the y or "b"-dimension of the conventional unit cell. For cubic symmetry we expect the x, y, and z unit cell dimensions to be equal

Now the tricky part. Here we'll try to find scattering from some other (hk0) planes for the NaCl and LiF crystals. To do this we must rotate the crystal mount (i.e., the slave plate) to allow for the correct scattering geometry. For scattering off the (hk0) plane we must rotate the slave plate by an angle ϕ which is equal to the angle between the (100) and (hk0) planes. (In Section D.25 of the manual this angle is referred to as angle SPQ). The angle between lattice planes (hkl) and ($h'k'l'$) is given by

$$\cos\phi = \hat{n}_{hkl} \bullet \hat{n}_{h'k'l'} \tag{2}$$

where, for a cubic lattice, the unit vector normal to the (hkl) plane is given by

$$\hat{n}_{hkl} = \frac{h\hat{x} + k\hat{y} + l\hat{z}}{\sqrt{h^2+k^2+l^2}}. \tag{3}$$

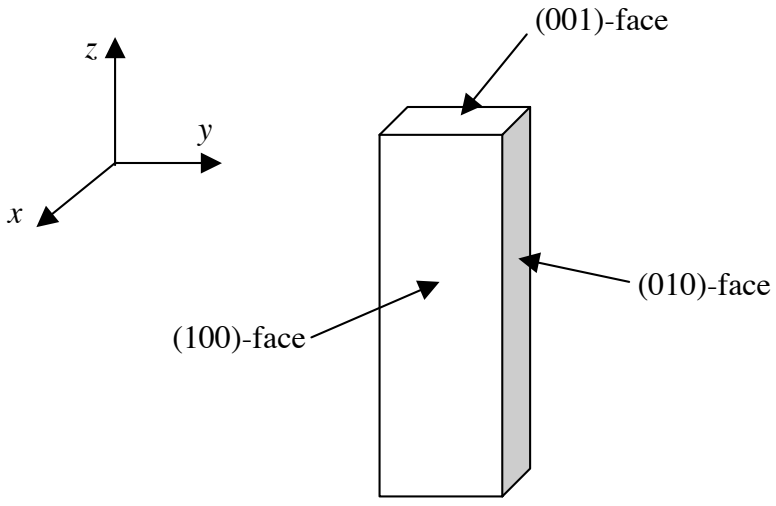
[Verify that Eqs. (2) and (3) give the correct angle between the (100) and (010) planes].

Before attempting to take any data, complete the prediction table given at the end of these instructions for both the NaCl and LiF crystals. In this table, ϕ is the slave plate angle and 2θ is the expected scattering angle for reflections from the (hk0) plane. Compute these expected

scattering angles using your Part 1 results for the lattice constant *a*. Now that you know what to look for, try to find it! Mount the crystal as for {100}-scattering and adjust the slave plate to the desired angle ϕ (counterclockwise rotation, noting start angle is ~0.5-1.0°). I had difficulty locating some of the (*nn0*)-reflections but, with some effort, was able to locate the other expected reflections (although some peak count rates were ≤ 30 cps). The higher angle peaks tend to be easier to locate. In some cases you may need to shift the crystal sideways in the holder as shown in D25.10 (the crystal needs to be offset away from the X-ray tube). If you can't find any peaks, try tweaking the slave plate angle ... the whole setup is extremely sensitive to the proper geometry (if the slave plate alignment is off by more than ~0.5° no scattering will be observed).

Data Analysis:

Your results for Part 1 will include I(θ) vs. 2θ plots for each data set and a table of scattering peaks for each crystal. Calculate lattice constants (with uncertainty estimates) for all four crystals and compare your results with literature values. Also determine the Miller indices (i.e., the *hkl* values) 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 listing the (*hk*0) reflections predicted and what was actually observed (include the peak GM count rate for each of these). Explain why some of the planes listed in the prediction table do not give scattering. For planes that do give scattering, how do you expect the scattering intensity to depend on the *hkl* indices? [Hint: The scattering intensity is approximately proportional to the *average number of scatterers per plane*]. Do you observe this expected intensity behavior? Finally, be sure to include a brief discussion of how the X-Ray tube and the GM detector work (see an intro or modern physics book). You can estimate the wavelengths of the K_α and K_β radiation for copper using H-atom energy levels with effective nuclear charge of Z–1. K_α and K_β correspond to n=2→1, 3→1 transitions, respectively.



Geometry of the large single crystals.

(*hk0*)-Scattering Prediction Table

In our original scattering geometry the normal to the (100)-face of the crystal defines the *x*-direction: $\hat{n}_{100} = \hat{x}$ (see above figure). For scattering off the (*hk0*)-planes we must rotate the crystal about the *z*-axis to align the normal to the (*hk0*)-plane with the *x*-axis. The required rotation angle is given by $\cos\phi = \hat{n}_{hk0} \bullet \hat{x} = h/\sqrt{h^2 + k^2}$. To predict scattering angles for these planes we must know the inter-plane separation d_{hk0} which is defined above with Eq. (1). Due to destructive interference within the unit cell, for an fcc-lattice we only get scattering from (*hkl*)-planes with *hkl* all even or all odd.

{*hkl*}-family	rotation angle **ϕ**	plane ***h k l***	plane spacing $\mathbf{d_{hkl}}$ (Å)	scattering angle $\mathbf{2\theta} = 2\sin^{-1}(\lambda/2d_{hkl})$
{100}	0°	200	_____	_____
		400	_____	_____
		600	_____	_____
{110}	45°	220	_____	_____
		440	_____	_____
		660	_____	_____
{210}	_____°	420	_____	_____
		840	_____	_____
{310}	_____°	620	_____	_____
{320}	_____°	640	_____	_____

Orientation of some crystal-plane families: