Solid State Physics: Experiment #2
X-ray Diffraction using the Transmission Laue 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. The present experiment is described on pg. 16 of the manual (D12 - Diffraction of X-rays; Laue). The goal is to produce an X-ray diffraction pattern on a piece of film from a small single crystal of LiF. The Laue method provides a nice demonstration of X-ray diffraction, however, quantitative analysis of the Laue pattern can be tricky (and there are much better methods available for structure determination). Thus we will be content with a primarily qualitative analysis in this case (although we will measure some scattering angles), saving the quantitative analysis for our next two experiments. **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.

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

1) Turn the power on key 90º 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 5 minutes. (If a crackling noise is heard when the X-rays are turned on, the tube 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. We will be working at 80 μA for this experiment (although the exact value is not critical).

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.

Experimental Procedure:

Follow the instructions for experiment D12 (pg 16). The LiF mini-crystals are inside the blue plastic sleeves in the vial labeled TEL 582.007. These crystals are very fragile ... HANDLE WITH CARE ... and not with your fingers.

The crystal should be mounted (with tape) directly over the aperture of the 1mm circular beam collimator. Before mounting the crystal, be sure you know how the collimator/plastic slide holder gets assembled and attached to the X-ray tube. Practice this without the crystal in place!

The X-ray film is in a blue box in the darkroom. There are two different filmpak types in the box. We're using the shorter one (filmpak 750/2). Load the film in the cassette labeled 562.013 and mount this in the slide holder after the latter has been attached to the X-ray tube.

I suggest placing the film in slot #3 and taking at least a 2 hour exposure. (This long exposure time is needed since our film is a bit old and thus has a very "fogged" background). Since the timer on the Tel-X-Ometer only gives up to about 55 minutes, you will need to reset it at least once during the exposure. You should also check the tube current periodically as it will float around. Try to keep it below, but near 80 μA.

After the exposure, remove the filmpak cassette and then very carefully remove the collimator/slide holder from the X-ray tube. Replace the crystal in its vial. Please do not leave these crystals out for days on end as they will pick up moisture from the air.

Film development:

The film can be developed any time after the X-ray exposure. Read the filmpak instruction sheet before starting! I suggest loading both the developer and fixer syringes before starting the development. Injecting these solutions can be tricky. You may want to play with one of the "spent" film holders sitting near the developing solutions. You may also want to insert a straightened paperclip into one of the injection ports just to make sure it's open. When you do inject the solutions, insert the tube to its hilt and hold the tube tightly to try and prevent solution from coming back up out of the port. Have a stopwatch ready for timing. Hold the filmpak over the red tray and inject slowly ... some solution will inevitably drip out ... its not toxic, but you'll want to wash you hands! (Sink is in the dark room). Scissors and small plastic tweezers are in the dark room. Use these to remove your film. Follow the film pack instructions to fix your film for permanent record. Although the film background is dark you should be able to clearly identify a diffraction pattern. Once dry, make some measurements and draw a quantitatively accurate blow-up of you diffraction pattern to be included with your write-up.

The Write-up:

This one should not be too long as there is only minimal data analysis. Here's what I want: An introduction to X-ray scattering. How X-rays are produced in the Tel-X-Ometer. A summary of your experimental procedure. Your results (i.e., the diffraction pattern and a set of calculated scattering angles). Conclusions (i.e., symmetry of crystal and anything else you can come up with). **Write-up due date: Friday, Feb. 14.**