

Appendix XVIII: Quick Start: The Tel-Atomic X-ray system (TEL-X-OMETER)

A. Background

1. The detector is a Geiger Müller tube. Note that a GM tube is essentially a proportional counter operated with the voltage high enough to be in the Geiger region. (The Mössbauer experiment uses a G-M tube operating in the proportional range.) This means that the voltage is high enough to generate a saturating avalanche of electron multiplication from the primary ionizing radiation. Thus the charge pulse is relatively independent of the amount of ionization produced by the primary radiation--in contrast with the proportional region.] For this tube, the Geiger mode is reached at the voltage of 350V.
2. Review the Radiation Safety manual and all lab instructions. This equipment has interlocks to prevent operation when the cover is not properly closed. It is also equipped with a timer. Set the timer to 30-60 mins of operation. Turn the key switch on to start the tube filament. Start the X-ray emission by pressing the red button on the diffractometer. If the red light in the button and the red light next to the tube do not stay on, one or more interlocks is not satisfied. Check and adjust the cover position and the timer position.
3. Check tube current with the multimeter (system starts at $\sim 85 \mu\text{A}$, and then settles over 10+ minutes to $\sim 70 \mu\text{A}$).
4. Turn on the high voltage and the electronics in the power supply for the Geiger Tube (voltage should be at 350V). Note that the output pulse is 15V and needs to be attenuated by 13 dB to 5V for input to the Mac digital counter (the attenuation is built into the cable between the G-M tube power supply/electronics and the Mac input).
5. Turn on the power switch to the power unit which supplies +24 V to the stepper motor driver.

B. Turn on the Mac and go to the data acquisition and control program:

- 1) Turn on the Mac or wake it up. Then click on the "Advanced Labs" icon.
- 2) Click on the "x-ray" icon within the advanced labs box. This will bring up an empty graph with various buttons to click on....

C. Initial Position:

1. **Check carefully that the sample rotation angle θ is exactly half of the detector arm position 2θ . Actually, it will be convenient to set the 2θ angle to exactly 30° . Then the two red lines on the sample mount ring should read 14° and 15° .** If they are close, then check the calibration by measuring one or two peaks for NaCl – if these do not concur closely with what is expected, return to $2\theta = 30^\circ$, should open the cover, remove any sample, and back off the knurled ring until the sample mount with the chamfered pin can be rotated. Rotate until the correct θ is obtained and retighten the ring. (If the $\langle 200 \rangle$ peak at 31.7° for NaCl is much below 1000 counts per second, then this alignment is probably off.) Readjust if

necessary. *It will be necessary to recheck this setting periodically during the lab period since it may drift away from calibration!!*

2. **Remove backlash and reset to the start position!** Set the 2θ angle slightly below the required initial angle by first entering a number in the “Delta two theta” box and then clicking on the Mac “manual adjust” button to control the stepper motor. Note that a negative number will cause motion to smaller angles and a positive number can be used to approach the start value in small steps.

D. Start a Scan

1. Change entries in the parameter menu as required by selecting the entry box using the mouse, typing the desired parameter and clicking to record the entry.

START ANGLE	initial 2θ angle in deg
FINAL ANGLE	final 2θ angle in deg
STEP SIZE	increment size in deg
INTEG TIME	time in ms to count at each position

2. Click on “start scan” and the data acquisition and scanning will begin. The data will be plotted live. Note that the default is to use autoscaling for the vertical scale. You may want to set the limits manually to 0 and 2000 to start.

Note: because of possible slippage of the $\theta / 2\theta$ alignment, it is best to do the first scan with no slits in place in front of the detector.

D. Save Data **(DATA ARE NOT AUTOMATICALLY SAVED!!)**

1. At the end of the scan, click on “save data.” Please save first to the hard drive using a file name of your choice. Also generate a hard copy of your screen by clicking on the “file” pull-down menu and clicking on “print screen.” Note that the printer is quite slow. Don’t give up; it will come! Meanwhile, you may go on to take more data. It will print in background.

Warning! If the detector arm is in danger of crashing into the latch towards zero degrees or the x-ray source towards $2\theta = 120$ degrees, just turn off the 24 V supply to the stepper motor. This also works if you mis-program the system!

- E. Before you leave the lab, copy your data onto your personal floppy disk for security and for off-line processing.