

Operating Procedures for the UGA Geology Bruker D8 Advance X-ray Diffractometer

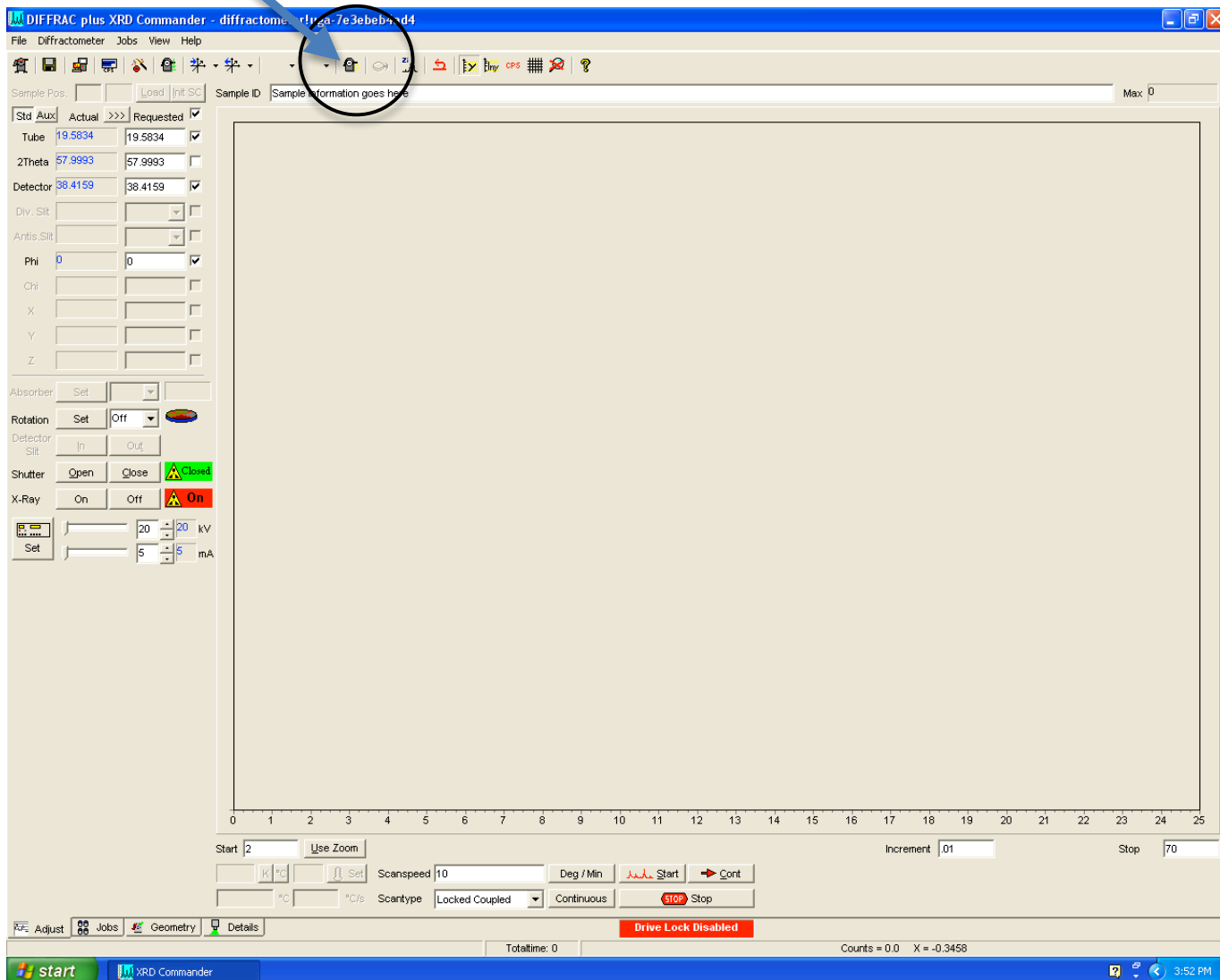
Start-up (X-ray warning lights on top of cabinet are on and water chiller is running)

This is the state that the diffractometer should be most often found. Check water chiller gauges for operation in proper ranges (Pressure <80 psi: Temp <80° F). Also look to see if there any puddles in and around water chiller and XRD unit. Leaks happen! Check the level of the water in the cooler by removing the gray lid and white cover. Level should be above clear plastic float on left inside of water tank.

At computer, open “Commander” program from the icon on computer screen.

In upper part of left panel of the Commander display, number should indicate position of tube and detector. Red numbers mean only an approximation. Clicking “requested” will put check marks next to the moving parts. The previous user should have left tube at 19.5834° and detector at 38.4159°. Look to see if the goniometer parts are actually in those approximate positions.

Select the “initialize” icon at top of screen. Tube and detector should shuffle and down as the machine initializes to specific positions near ~20° for tube and ~40° for detector.



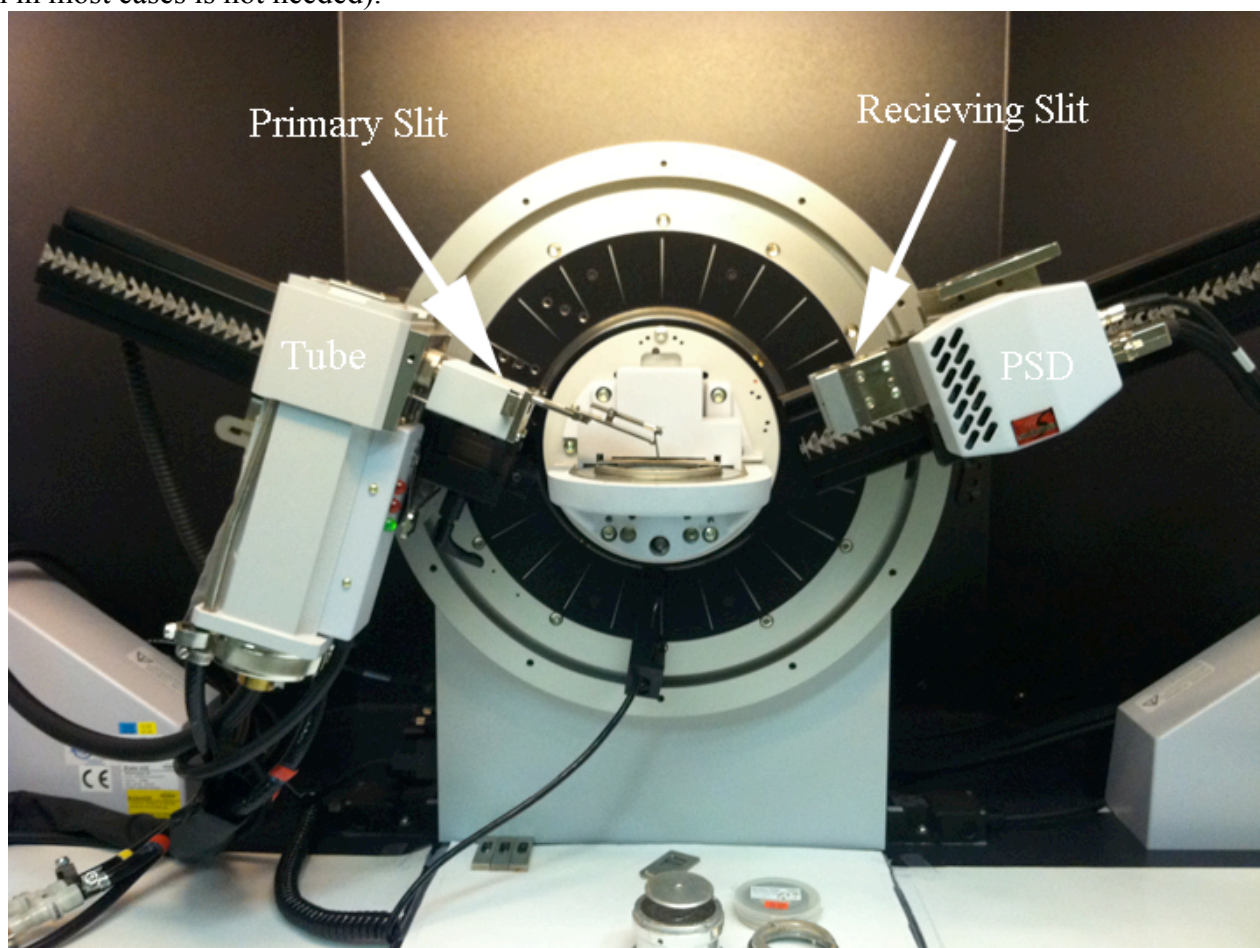
If initialization is unsuccessful, stop and seek help.

Bring power up to 40 mA and 40 kV by changing settings at bottom of left panel and selecting “SET”. The machine will ramp up to new settings at its own pace and does not require you to make incremental increases.

Wait five minutes for system to warm, if the system has not been used in the last hour.

Your first run should always be the corundum standard (NIST SRM1976), with scan settings for Start = 40°, End = 42°, Step increment = 0.01°, and Rate = 0.1 sec/step. Corundum should have a peak at 41.05 °2 θ using Co K_{α} radiation. Once scan is finished, use the screen cursor to find the peak position and intensity of the largest peak. Save this scan as a file in your folder and give it a unique name like Cor_XX_XX_XX, where XX_XX_XX = the current date (e.g., 02_10_12 for February 10, 1012).

Record in the log book your Name: Date: Start time: Project Name: and the values for the corundum peak position and intensity. Check the values to be sure they are similar to those previously recorded (41.05 °2 θ and over 2500 counts per second with the 0.6 mm slit). The intensity may vary depending upon the primary slit installed. Check the primary slit, which is shown in the position labeled in the image below. 0.6 mm is a good compromise if you do not know where to start. Intensity may also be lower if you have a receiving slit installed (which in most cases is not needed).



You will be reminded later on, but obviously record the End time: when you finish.

Loading a sample

If the green light on the tube is on, the shutter is shut and no X-rays are being emitted (the desired case for sample insertion).

Press the green “open door” button on one of the two side panels to unlock the glass doors.

There are several styles of sample holders. Choose the one appropriate for you.

Your first run should have already checked the machine calibration.

After putting your sample in the appropriate holder, fit the holder into the machine’s sample mount by inserting from below until a magnetic attraction locks the sample holder in. Close doors and make sure to “jiggle” the black handles click into the closed position.

Do not leave the doors open for more than about five minutes!! If they are left open for extended periods of time a safety interlock will be tripped and the machine will become disabled. If this happens, then you ‘ll need to find an experienced user that knows how to clear the interlock registers.

Scanning

At the bottom of the commander screen, enter in starting and stopping $^{\circ}2\Theta$ values. If you do not know what to enter, then use 2° and 70° as default start and stop values, respectively.

Between those two, set increment (step) by which machine advances in degrees (typically well less than $1^{\circ} 2\Theta$). If you don’t know what to enter, then use 0.02° as a default value

Set seconds per step (or you can select that label to change to degrees per minute). These four inputs (start, stop, increment, and seconds per step or degrees per minute) will determine how long it will take to run your scan. If you do not know what enter, then use 10 DEG/MIN as the default value.

Scan type is typically set to “locked coupled”.

Check your inputs again – sometimes Commander resets them.

If you want your sample to be rotated during the scan, set rotation speed (in revolutions per minute) near middle of left panel. Caution: Low angle scans ($<15^{\circ} 2\Theta$) will have some intensity blocked by the edges of the sample holder. If do not want this to happen, then align the notches in the holder such that they open toward the tube and detector. Otherwise, if you want to spin your sample, then use 15 RPM as the default.

Hit “Start”. The “Busy” light on right side of machine should come on.

If you are performing a quick reconnaissance scan, and then you want higher quality data, you can select a region of your reconnaissance pattern and hit “use zoom” below to input that region as the start and stop of a next, more detailed, scan. Modify the scan step increment and rate as desired for a more detailed scan.

Once your scan is complete, there is a comment line on the top of the pattern on the screen. Enter details about the sample and any other relevant information (you may want to do this before you start the run).

Save your pattern, if desired, using “Save as” in the standard Windows menu at upper left of computer screen. Save to an appropriate location within the folders for class work, students, faculty, et al. Commander will customarily save in a “.raw” format. All data should be saved in the C: drive User_Data folder, in a sub-folder with your name.

EVA and TOPAS are applications with which to manipulate data. Start with using EVA. Use “open” or “import” to access your pattern. Use the toolbox then to work with the pattern, choosing tasks in the tool box and using the cursor to outline areas of interest on the pattern. See manuals, help menu, or ask a knowledgeable XRD user how to process data. At this point, there are an infinite number of things you can do to process your data and it is beyond the scope of this “cheat sheet” to lead you through such details.

When you are done: Leave in Stand-by mode

Record End time in log book.

In Commander, set power down to 20 kV and 5 mA and hit set.

Select the initialize icon. Clean Up sample holders and other powders left around the lab.