

Standard Operating Procedure: Bruker D8 Discover X-Ray Diffractometer

(You MUST be trained by an authorized BSCMC trainer to use this instrument)



Hazards: This unit produces a high intensity X-ray beam. Take all precautions to avoid exposure to the x-rays.

Warning: Beryllium! Do not touch the front window of either detector as they both contain Beryllium. Fumes of the dust from Beryllium and its compounds can be hazardous if inhaled!

Typical Experiment Sequence:

1. Sign in on the electronic calendar
2. Visually check the diffractometer for instrument status.
3. Open the cooling water valves and turn the x-ray generator ON.
4. Launch the **D8Tools** software to check the system status and ramp the power up.
5. Launch the operating software:
 - a. **GADDS** for use with the HI-STAR area detector
 - b. **Commander** for use with the Scintillation detector.
6. Check the x-ray beam intensity
7. Load and align sample
 - a. Use the **Laser-Video System** with **GADDS** and the HI-STAR area detector
 - b. Use a Z-scan and half intensity method or the **Laser-Video System** with **Commander** and the Scintillation detector.
8. Set up scan and save data.
9. Remove sample, reduce power, turn the generator off and shut off the cooling water
10. Sign out on the Sign electronic calendar Be sure that the calendar accurately displays the number of hours the XRD was OCCUPIED by your experiment.

Procedures for ALL X-Ray Diffraction Experiments on the Bruker D-8 Discover:

1. Sign in on the electronic calendar at <https://boisestate.ilabsolutions.com>

Visually check the diffractometer for instrument status. Check the machine status on either left or right column before using the machine. The “Ready” and “On” lights should be on. There might be an “alarm” due to the x-ray generator being OFF.



a. Left Control Column:

- STOP button:** If hit it switches off the control electronics. The X-Ray source is turned off and all moving drives will stop instantly. The stop button should only be used in emergency situations and not for normal shut down of the diffractometer system.
- Light button:** This button turns ON/OFF the fluorescent tube located at the ceiling of the enclosure.
- Fan button:** This button turns the fan located on top of the enclosure ON or OFF.
- System Instrument-Key Switch:** This key will lock/unlock the system. During normal operation the switch must be in position “unlocked”.
- Open Door button:** This button must be pressed to open the front door. If the Open Door button is activated while the tube window is open the X-ray shutter will close automatically and it will disrupt the experiment.
- Ready light:** Status of High Voltage Generator. Illuminated orange display indicates that the high voltage generator is operating. Flashing display indicates the high voltage generator is ramping up.
- On light:** Illuminated green display indicates the control electronics are ready.
- Alarm light:** Illuminated red display indicates a pending system alarm, flashing red light indicates a pending warning.
- Busy light:** Illuminated yellow display indicates that a measurement is in progress. Flashing yellow display means that the system is not ready.

b. Right Control Column:

- Stop button:** Functions the same as the Stop button on left column.

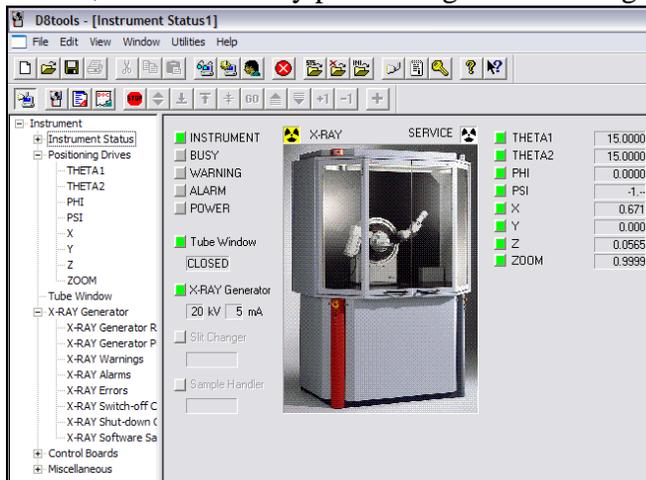
- ii.  **Power OFF button:** It switches off the control electronics, the high voltage generator and all components connected to the AC outlets.
- iii.  **Power ON button:** It switches on the control electronics, the high voltage generator and all components connected to the AC outlets.
- iv.  **High Voltage- Rotary Switch:** Used for turning high voltage on or off.
- v.  **Open Door button:** Functions the same as the button on the left column
- vi. **Ready, On, Alarm and Busy lights** have same functions as those on the left column.

3. Starting your session

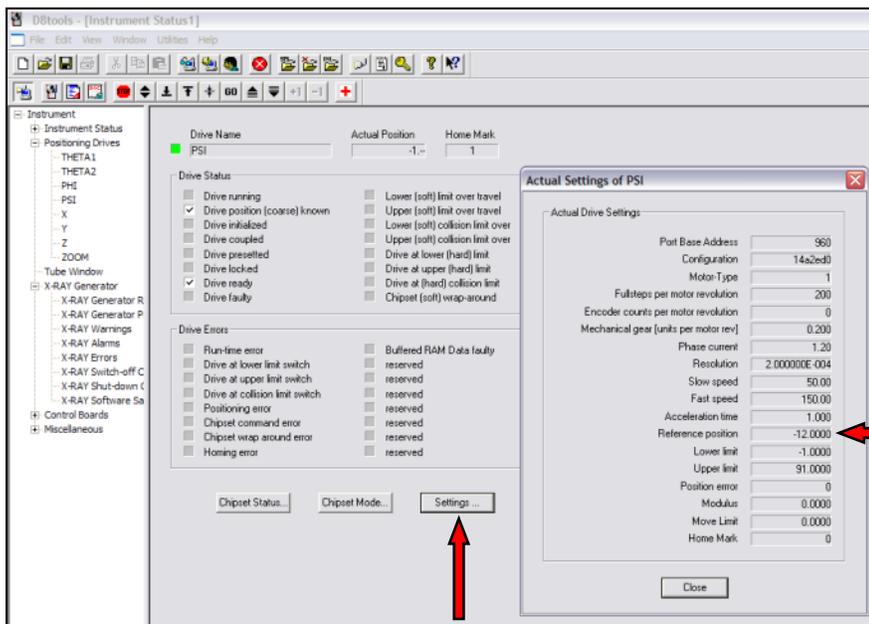
- a. Open the cooling water valves (on the wall behind the instrument) and check for any leaks.
- b. Turn the x-ray generator ON by flipping the HV switch clockwise for few seconds and this will automatically set the power to 20kV and 5 mA.
- c. Open the D8 tools and check the system status.
(→Online Status, →Online Refresh)
- d. Check for any alarms
 - i. If they appear, click on the red or orange box to be directed to the cause
- e. Ramp up the X-ray power(→X-Ray Generator, →Utilities, →Set kV/mA).
 - i. Set 30 kV, 10 mA and wait 2-3 minutes
 - ii. Set 40 kV, 15 mA and wait 2-3 minutes
 - iii. Set 40 kV, 30 mA and wait 2-3 minutes
 - iv. Set 40 kV, 40 mA
- f. Check the status of the drives
 - i. Are the drives initiated? (Make sure the drives have significant figures (i.e. 23.047 vs. 23.---))



- i. If not, initiate them by performing the following:



- a. Click on the green box next to the drive to be initiated, then check the settings of the drive (→Settings)
- b. Check the reference position of the drive



- c. Then physically look at the drive to determine which way it needs to go in order to be at the reference position
 - i. If it needs to go in the positive direction to fulfill the reference position, push the find reference up button. Likewise, if the drive needs to go in the negative direction, push the find reference down button (→ )

NOTE: Be sure to have the mouse on the STOP icon to be able to stop the drive if it looks as though it will crash (this will require you to watch each drive as it is initiated!)

- d. Once the drive has stopped moving, push the adjust button (→ )
- e. Repeat steps a. and b. for any of the drives that need to be initiated.
- ii. Do the drives appear to be in the indicated position? (Look at the values in D8 Tools for θ_1 and θ_2 and compare them to the physical locations of θ_1 and θ_2 on the goniometer).
- g. Close the **D8Tools** software.

Procedures for X-Ray Diffraction Experiments using the point detector:

4. Launch the operating software:

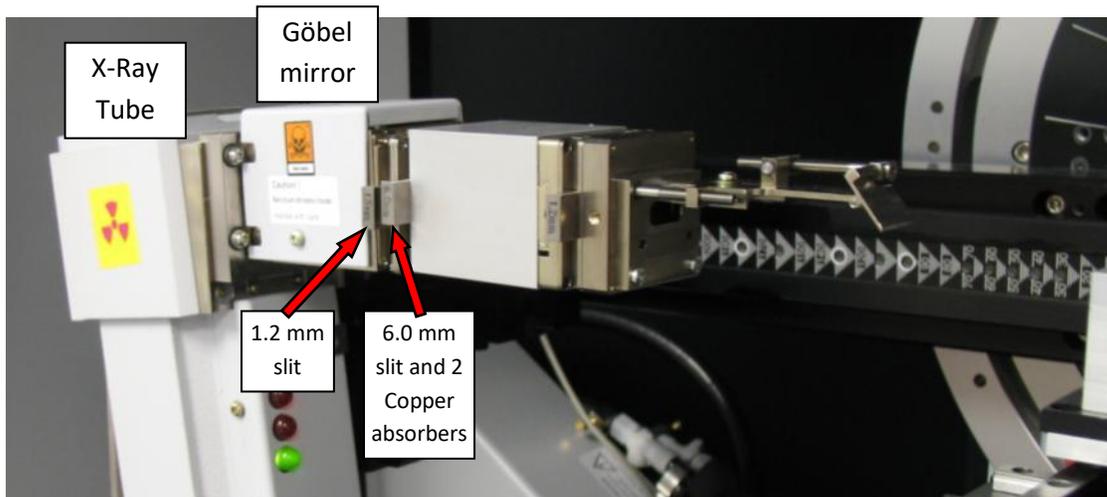


- a. Open the **Commander** program for use with the point detector

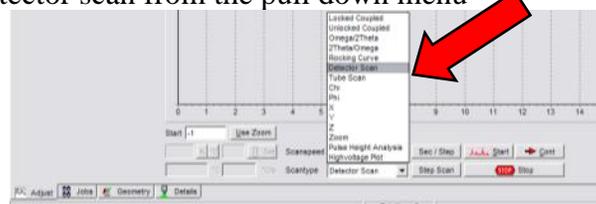
5. Check the x-ray beam intensity (Make sure there is no sample or sample holder that will interfere with the path of the x-rays)

NOTE: It is imperative that the copper absorbers are in place! If not, the detector could be damaged!

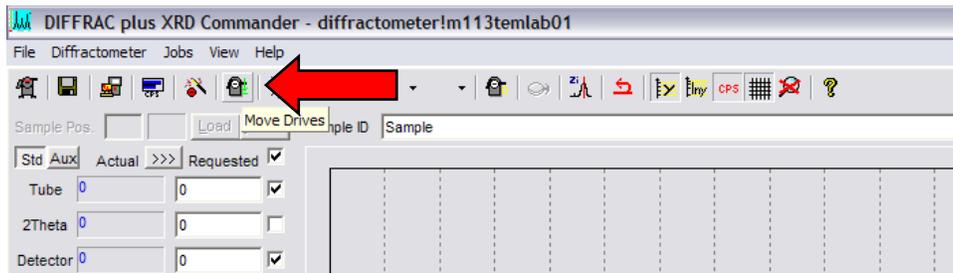
- a. Make sure the following slits are in place:
 - i. 1.2mm slit in the Goebel mirror exit slit (furthest left)
 - ii. 6.0mm slit with the TWO copper absorbers in the next slit



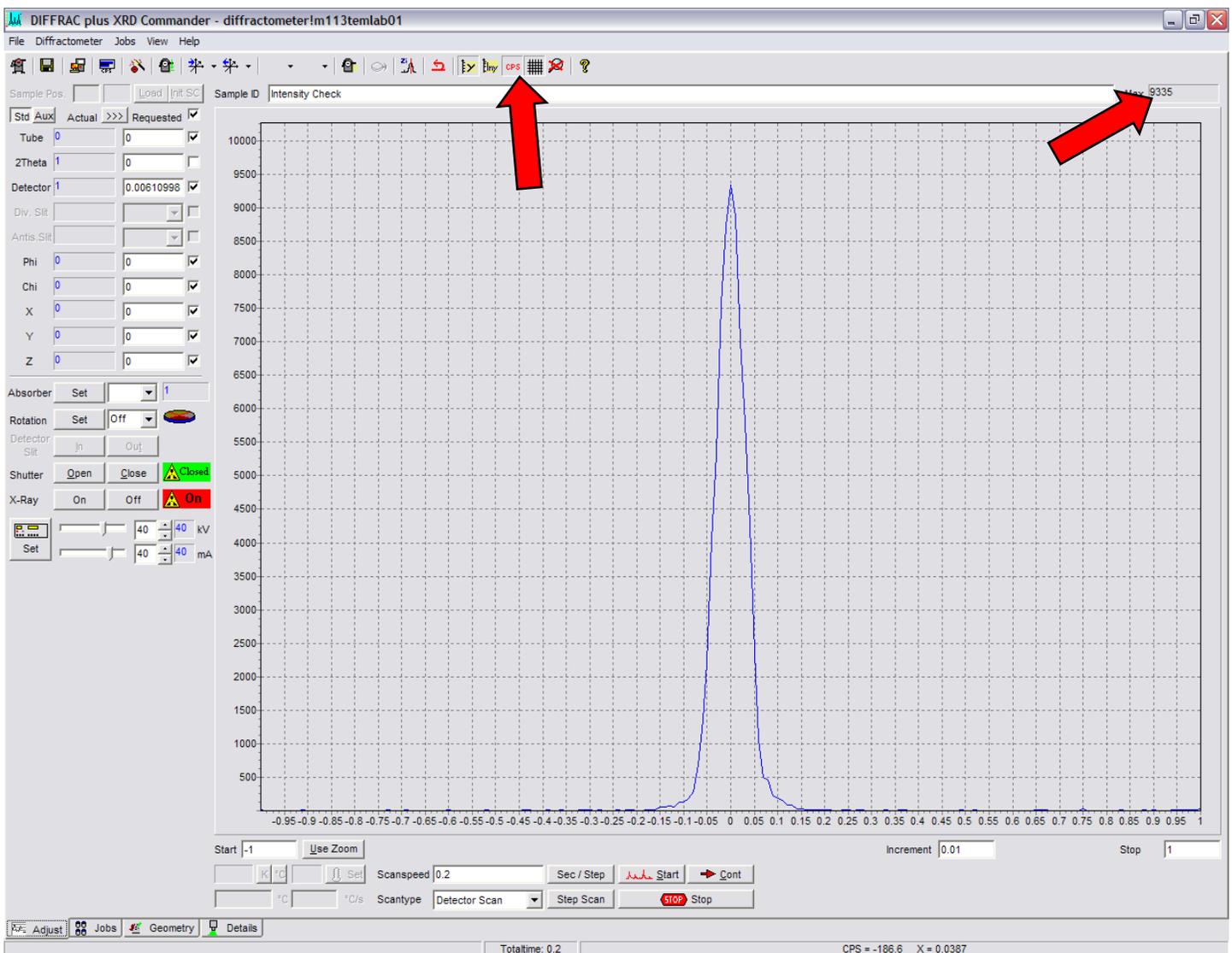
- b. Set up a detector scan from -1 to 1 degree
 - i. Select Detector scan from the pull down menu



- ii. Make sure that all of the drives are at 0
 1. Type a 0 in each drive command window and then click on the Move Drives icon to move the drives to 0 position.



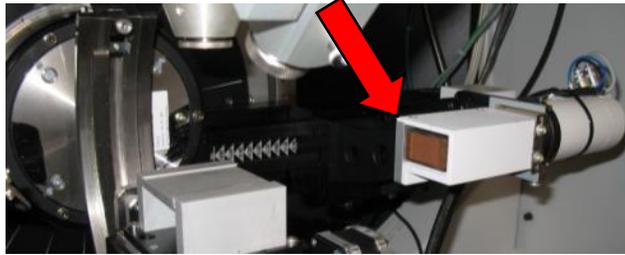
- iii. Make sure the y-scale is in CPS
 - iv. Set the increment to $0.01^\circ/\text{step}$ and the scan speed to $0.2 \text{ sec}/\text{step}$
 - v. Press the  icon.
- c. Make sure that the max count rate is 65000 CPS or more. If not, please contact an authorized BSCMC trainer.
 - d. Make sure the peak is approximately centered about 0. If not, please contact an authorized BSCMC trainer.



6. Load and align the sample (Depending on the sample and the stage, this can be done using the **Laser-Video System** or using a **Z-scan**)

NOTE: Things to be careful of when loading your sample:

- Do not touch the sollar slits



- Be gentle with the laser/video system
 - These are precisely aligned to give you “good” data
 - Do not bump the X-ray optics
 - A very slight bump could easily result in a loss of 30% of your beam intensity
- a. Open the doors to the XRD (Push **Open Door** button on either side of control column, then open the window.)  or 
 - b. Put your sample on the XYZ table (stage will depend on preference, special instruction may be required) and position it directly under the camera.

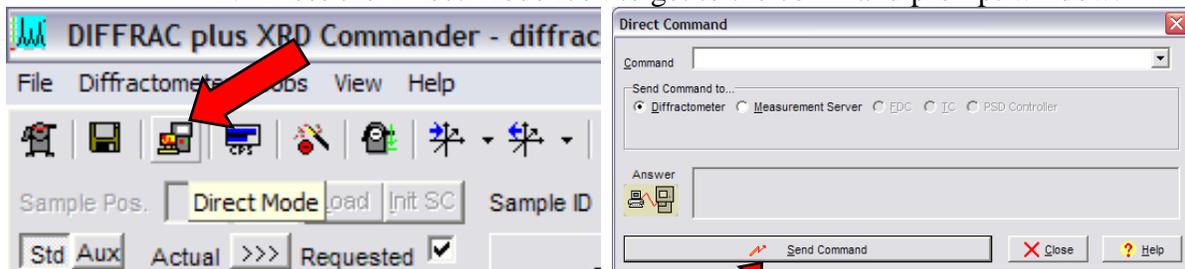
6.a Align the Z-height using the Laser Video System

- i. Align the sample to the instrument using the **Laser-Video System**

NOTE: All of the components including the X-ray beam, camera, and laser have been aligned to aim at a point which is called universal center of the goniometer. To align your sample surface at this universal center, you need to use the **Manual Controller** to move the XYZ table.

- ii. Activate the **Manual controller** and turn the laser on:

1. Press the Direct Mode icon to get to the command prompt window.

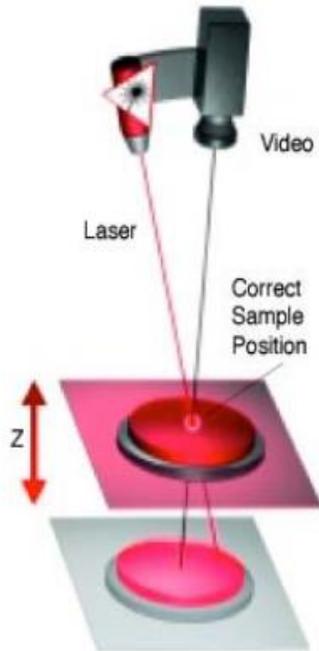


2. Type “RC1” and press “Send Command” to give the computer control.
3. Type “OC16,1” and press “Send Command” to turn the laser on.
4. Type “RC0” and press “Send Command” to give command to the manual controller.
5. Position the stage and sample accordingly.

- iii. Understanding the **Manual Controller functions**. The drives used are as follows (the numbers on the controller correspond to the following drive numbers):

1. Θ_1 – X-ray source (aka tube)

2. Θ_2 – Detector (In this case the HI-STAR area detector)
3. Φ (phi) – Rotation of the sample about the z-axis
4. χ (chi/psi)– Rotation of the sample about the Eulerian cradle
5. **X**
6. **Y**
7. **Z**
8. **Camera Zoom** ($1X \leq \text{Zoom} \leq 7X$)

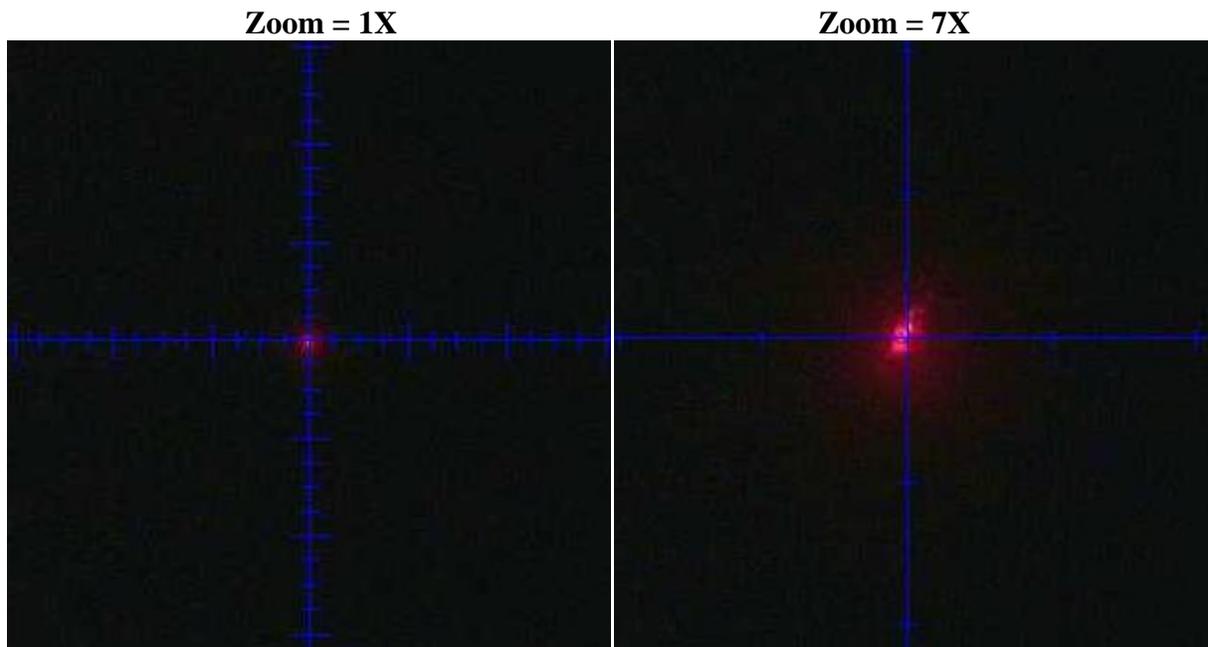


- iv. The Manual controller has two functions:
 1. **Command** (set point) mode (access by pushing the “F1/DRVC” button on the controller)
 - a. Enter three commands (Drive #, Instruction, Location)
 - i. **Drive #** is where the drive you want to move is called on
 - ii. **Instruction** is what you want the drive to do (“Go To” is always the number 2)
 - iii. **Location** is where you want the drive to move to
 - iv. (i.e. if you want the video zoom to go to 7X, you would type in “8,2,7” and then press “ENTER”. The zoom will automatically go to 7X)
 2. **Manual** (video game) mode (access by pushing “SHIFT” then “F1/DRVC” on the controller)
 - a. Identify the drive you want to move (1-8, see above)
 - b. To change the position of the drive, use the arrows (\downarrow or \uparrow).
 - c. To change the speed of movement of the drive, use the “+” or “-“ buttons.

!!!Warning: Make sure that none of the drives will run into each other when they are moved since there are NO safety switches installed for this purpose. The “Emergency Stop”   buttons can be used if needed but it is preferred to use the **ESC/STOP** button on the controller if you see a potential problem.



- v. Open **VIDEO** program, Turn on the light in the XRD chamber as needed 
- vi. Zoom the camera out (zoom = 1) to help easily find your sample in the video window.
- vii. Move the X and Y drives to the area of interest on your sample (using the controller in manual mode).
- viii. Move Z to focus the laser on your sample (The laser spot should be in the center of video window)
 1. The laser might be difficult to see on reflective samples. If this is the case, move Chi/psi to 22.5 degrees (splitting the angle between the laser and the video) to cause the laser to reflect back into the camera.



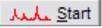
!!! Warning: Always leave one finger on “**ESC/STOP**” button when you are using command mode. Press “**ESC/STOP**” button immediately if the moving part is going to hit other part.

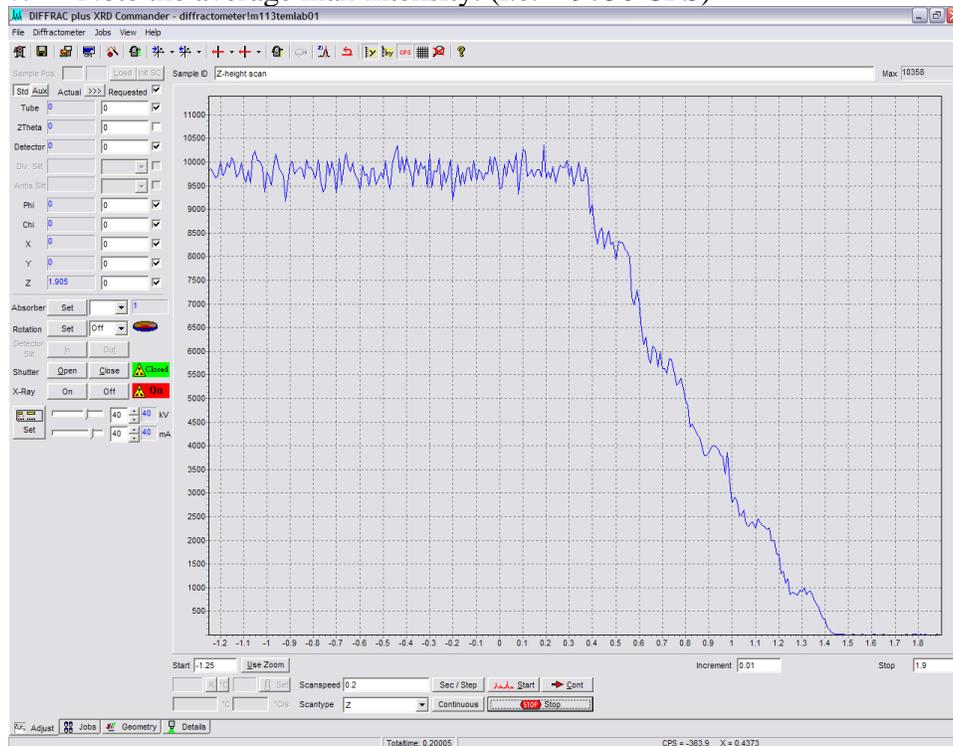
- ix. Once your sample is in focus and the laser is in the center of the video window (centered on the crosshairs), zoom the camera in (zoom = 7) to fine tune your sample position.
- x. Once the laser spot is centered, the sample is aligned and ready to take data (return chi/phi to 0 (if applicable)).

!!!Warning: You can use the controller to move Θ_1 and Θ_2 to the desired 2θ angle. Make sure the detector will not get hit by the direct beam (as a rule of thumb, always keep $\Theta_1 + \Theta_2 > 15^\circ$).

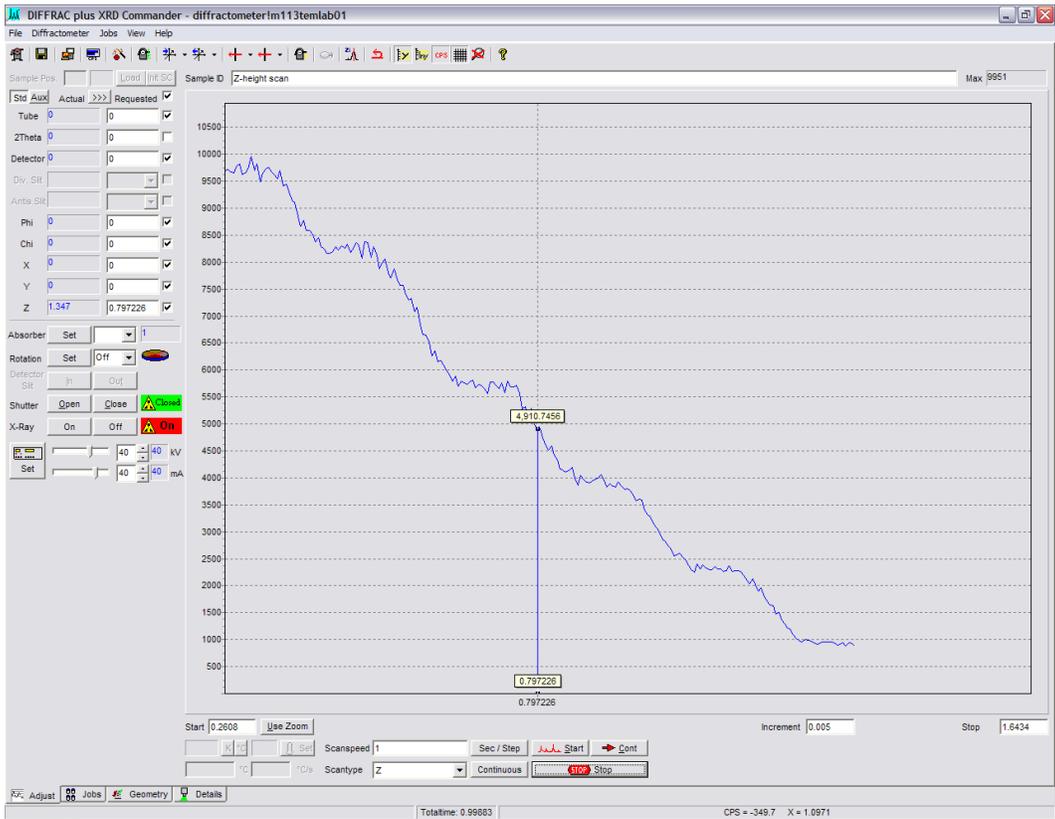
1. Back in the Direct Command window in Commander, type “RC1” and press “Send Command” to give control back to the computer.
 2. Type “OC16,0” and press “Send Command” to turn the laser off.
- xi. Exit the Direct Command window by closing the prompt window.

6.b Align the Z-height the Z-scan half intensity method.

- i. Align the sample to the instrument using a Z-scan and half beam intensity method.
- ii. In the **Scantype** drop down menu, choose “Z”
- iii. Set the scan at -1.25mm and end the scan at 1.9 mm
- iv. Set the Increment to 0.01°/step and the Scanspeed to 0.2^{sec}/step and click .
- v. Note the average max intensity. (i.e. ~ 9750 CPS)



- vi. Typically run a fast scan to start, then zoom in on the area of interest, click .
- vii. Set the Increment to 0.005°/step and the Scanspeed to 1^{sec}/step and click .
- viii. Once the scan is completed, double click on the where the beam intensity is approximately one half of the maximum intensity. (i.e. 4875 CPS). Once you double click on the scan, the Z-position is automatically stored in the Z-drive command window.



NOTE: All of the components including the X-ray beam, camera, and laser have been aligned to aim at a point which is called universal center of the goniometer. To align your sample surface at this universal center, you need to use the **Manual Controller** to move the XYZ table.

7. Set up a scan and save data:

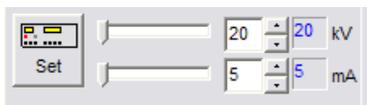
- Set up the scan that is of interest for your data.
- Unless you are using a script, Commander DOES NOT automatically save your data! Make sure that you save your data before you attempt to do ANYTHING further with the software!

8. Remove your sample

- You are responsible for removing your sample as soon as your scan is finished. **The Boise State Center for Materials Characterization is NOT responsible for any samples.** If your sample is left on the instrument, the next user has the right to remove the sample and set it aside. Clean up the stage and leave the instrument as clean, if not cleaner, than when you arrived to use it.

9. End of session

- a. Set the power generator to 20 kV and 5 mA by setting each scroll bar to the far left and pressing the SET icon. Make sure the generator reaches 20kV and 5mA prior to closing out of Commander.



- b. Close Commander.
- c. Wait for 5 minutes
- d. Turn the X-ray generator OFF by flipping the rotary switch counter clockwise for a few seconds (Do not shut the main power down)
- e. **Close the valves for cooling water.**

10. Sign out on the iLab electronic calendar

Be sure that the calendar accurately displays the number of hours the XRD was OCCUPIED by your experiment.