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 iLab 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. Be sure that the iLab 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 "On" light should be on. There might be an "alarm" due to the x-ray generator being OFF.

a. Left Control Column:
   i.  **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.
   ii.  **Light butto**n: This button turns ON/OFF the fluorescent tube located at the ceiling of the enclosure.
   iii.  **Fan button:** This button turns the fan located on top of the enclosure ON or OFF.
   iv. **System Instrument-Key Switch:** This key will lock/unlock the system. During normal operation the switch must be in position "unlocked".
   v.  **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.
   vi. **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.
   vii. **On light:** Illuminated green display indicates the control electronics are ready.
   viii. **Alarm light:** Illuminated red display indicates a pending system alarm, flashing red light indicates a pending warning.
   ix. **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:
   i.  **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

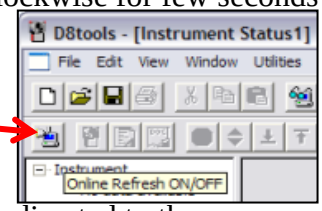
vi. **Ready, On, Alarm and Busy lights** have same functions as those on the left column.

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.

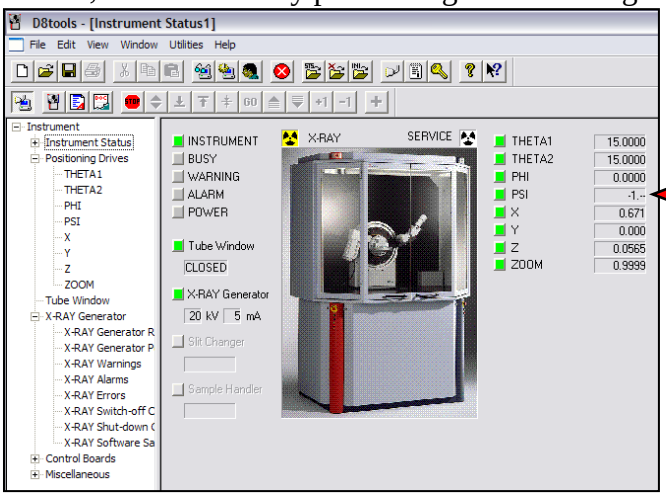
c. Open the D8 tools and check the system status.
(→Online Status, →Online Refresh)



d. Check for any alarms
(An alarm or warning will prompt a red or orange box)
   i. If they appear, click on the red or orange box to be directed to the cause

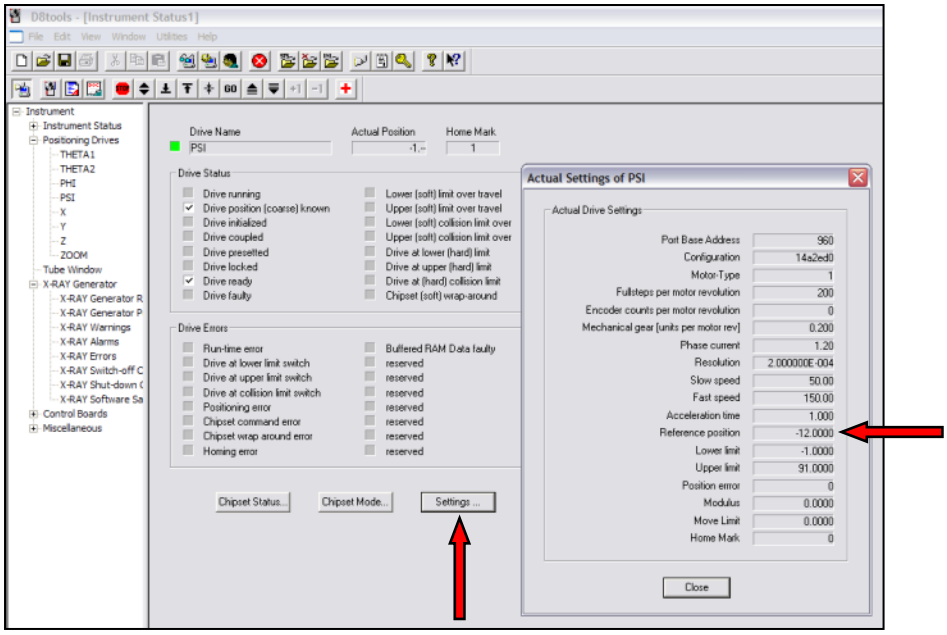
e. Ramp up the X-ray power(→X-Ray Generator, →Utilities, →Set kV/mA).
   i. Set 30 kV, 10 mA and wait 2-3 minutes
   ii. Set 40 kV, 15 mA and wait 2-3 minutes
   iii. Set 40 kV, 30 mA and wait 2-3 minutes
   iv. Set 40 kV, 40 mA

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



Needs initialized

      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.

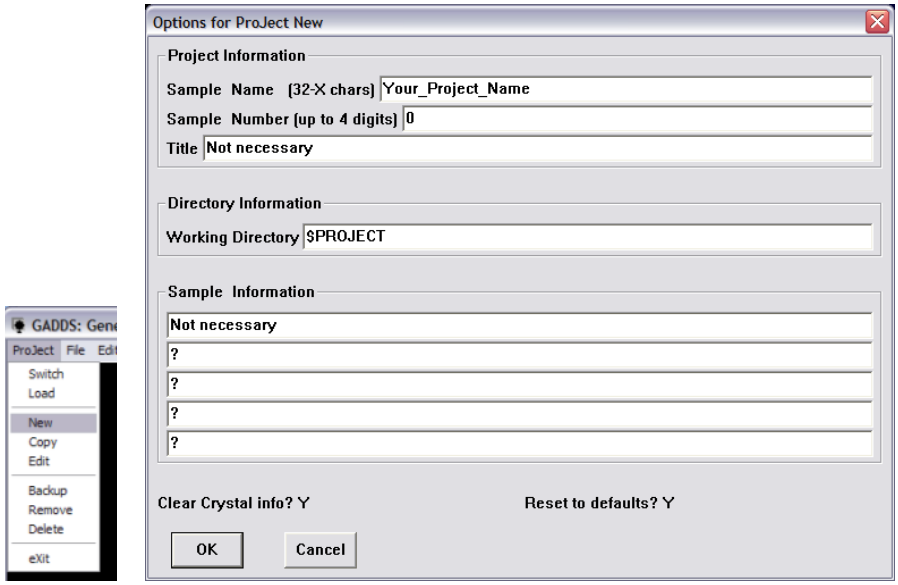
ii. Do the drives appear to be in the indicated position? (Look at the values in D8 Tools for θ_1 and θ_2 and compare them to the physical locations of θ_1 and θ_2 on the goniometer).

g. Close the **D8Tools** software.

Procedures for ALL X-Ray Diffraction Experiments using the HI-STAR area detector:

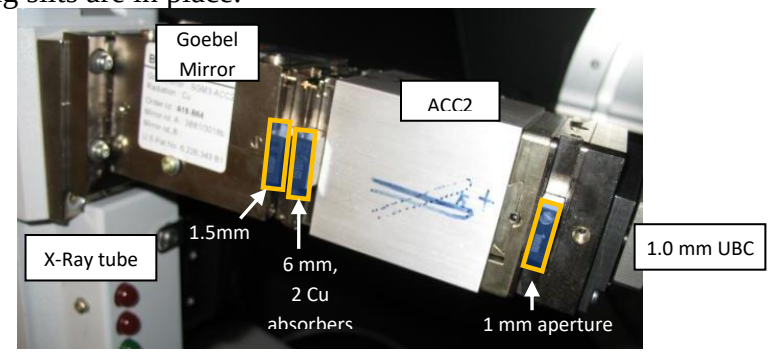
4. Launch the operating software:

a. Open the **GADDS** program for use with the HI-STAR area detector (It will prompt "set generator to user setting: 40 KV and 40 mA" →Click Yes)
b. Create a new project folder (→ Project, →new, input information accordingly. Use the working directory C:\Frames\"year"\"month"\"Sample Name" (This automatically saves subsequent data collections to the above file path))



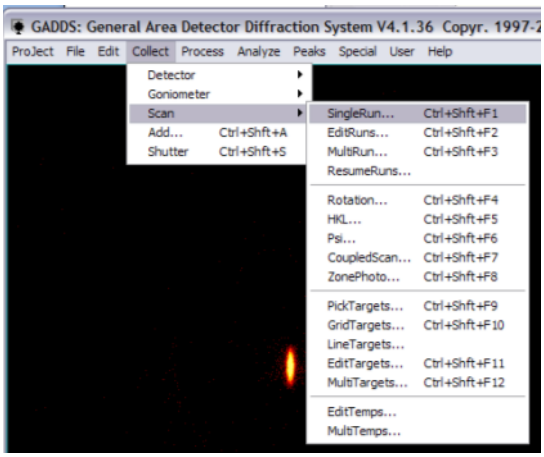
5. Check the x-ray beam intensity (Make sure there is no sample or sample holder that will interfere with the path of the x-rays)

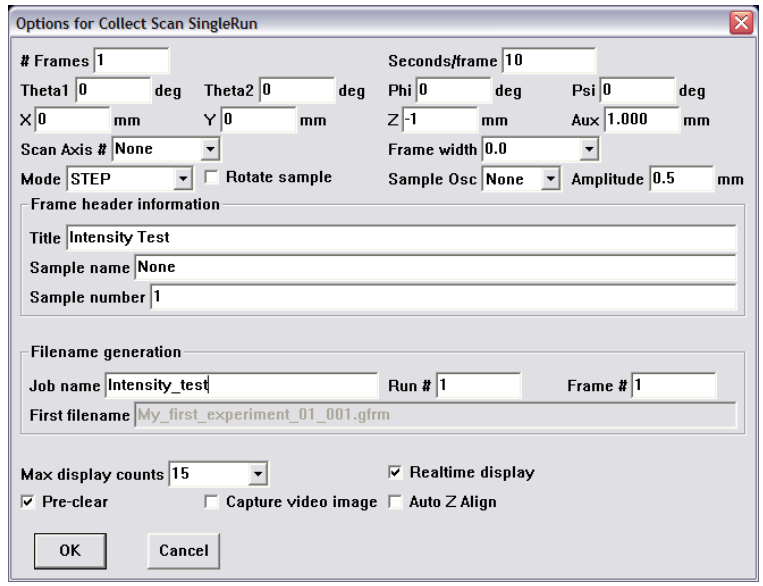
a. Make sure the following slits are in place:



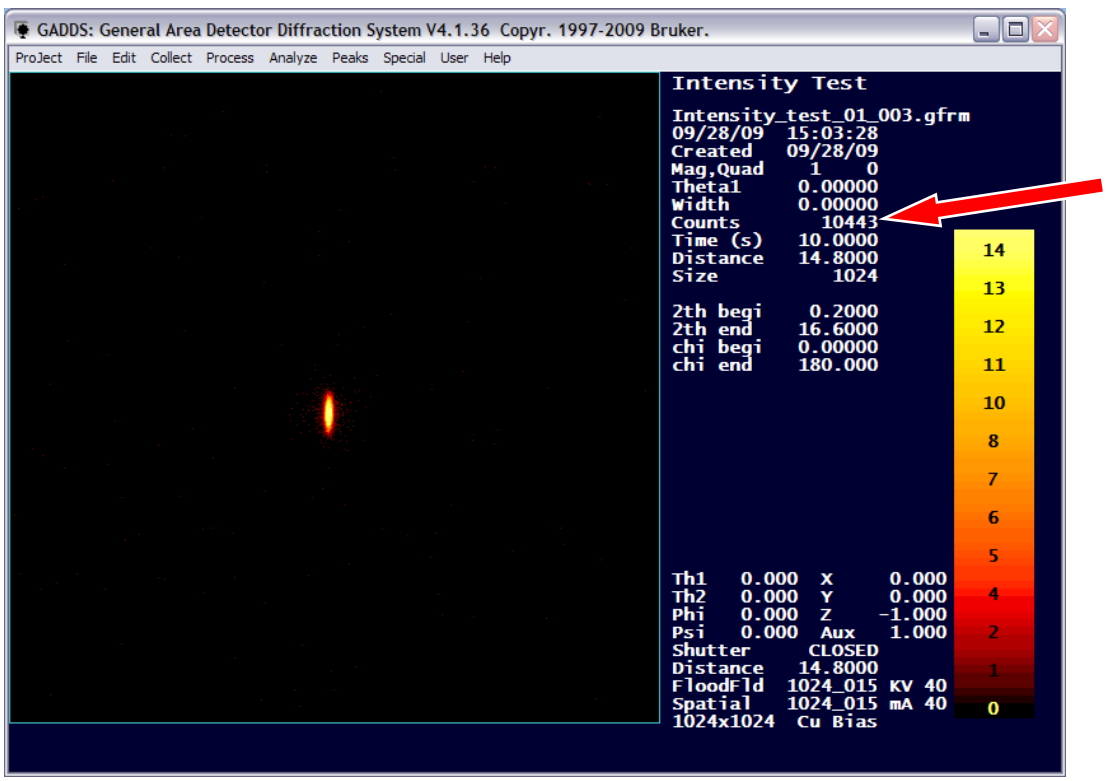
   i. 1.5mm slit in the Goebel mirror exit slit (furthest left)
   ii. 6.0mm slit with the TWO copper absorbers in the next slit
   iii. 1.0mm aperture in the ACC2 exit slit
   iv. 1.0mm Universal Beam Collimator is attached

b. Set up a 10 second scan of the beam directly into the detector
   i. (→ Collect, →Scan, →SingleRun)





c. Make sure that the detector receives around 6,000 counts or more. If not, please contact an authorized BSCMC trainer.



6. Load and align the sample (The **Laser-Video System** has to be used with the HI-STAR area detector)

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

- Do not touch the face of the detector
  - It is made of Beryllium and is hazardous
- Do not bump the detector
  - Could cause a short and cost a lot of money to fix.
- Be gentle with the laser/video system

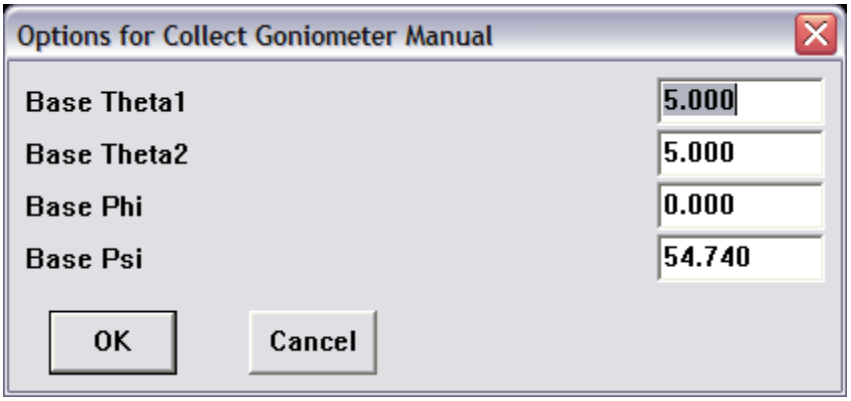
    - These are precisely aligned to give you "good" data
- Do not bump the X-ray optics
    - A very slight bump could result in a loss of 30% of your beam intensity

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

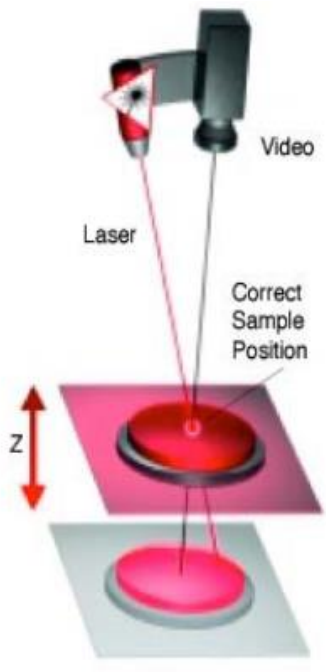
NOTE: All of the components including the X-ray beam, camera, and laser have been aligned to aim at a point which is called universal center of the goniometer. To align your sample surface at this universal center, you need to use the **Manual Controller** to move the XYZ table.

ii. Activate the **Manual controller** and turn the laser on:
    1. While <u>active</u> in the GADDS window. Press "**Ctrl+Shift+M**" to activate the manual controller. Press "**OK**" when the following screen appears.



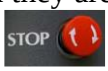
    2. Push "**L**" on the keyboard to turn the laser on.

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 Eularian cradle
    5. **X**
    6. **Y**
    7. **Z**
    8. **Camera Zoom** (1X ≤ Zoom ≤ 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)

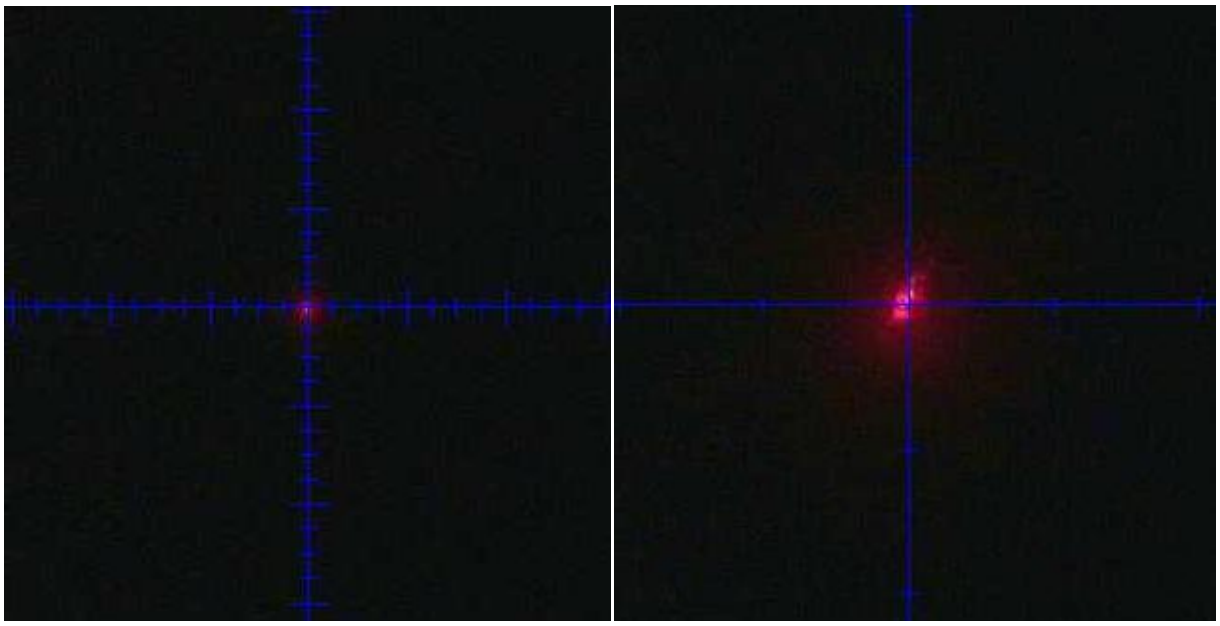
   2. **Manual** (video game) mode (access by pushing "**SHIFT**" then "**F1/DRVC**" on the controller)
      a. Identify the drive you want to move (1-8, see above)
      b. To change the position of the drive, use the arrows (↓ or ↑).
      c. To change the speed of movement of the drive, use the "+" 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 is 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.

Zoom = 1X **Zoom = 7X**



!!! 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.

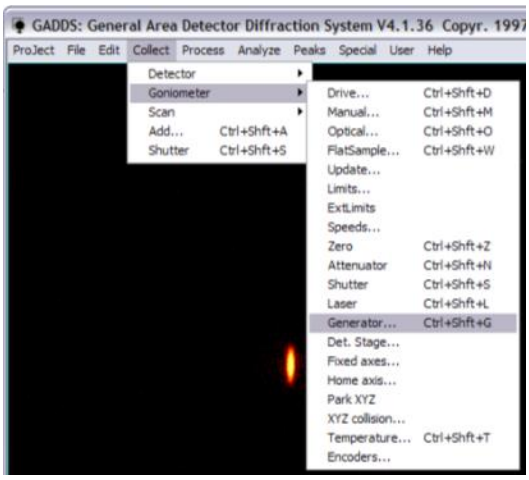
ix. Once your sample is in focus and the laser is in the center of the video window (centered on the crosshairs), zoom the camera in (zoom = 7) to fine tune your sample position.
x. Once the laser spot is centered, the sample is aligned and ready to take data (return chi/phi to 0 (if applicable)).
xi. Turn the laser off
    1. Type "**L**" in the GADDS program

!!!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 $\Theta_1 + \Theta_2 > 15°$).

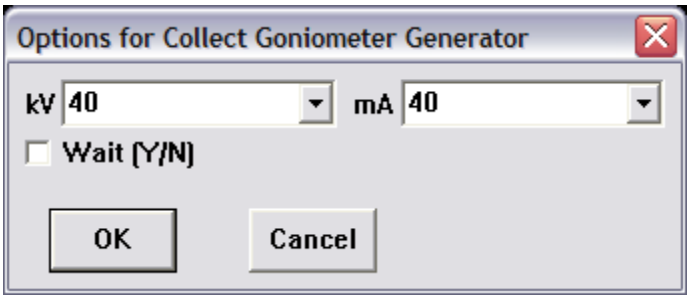
xii. Exit Manual control (→ press Esc.).

7. Set up a scan and save data:

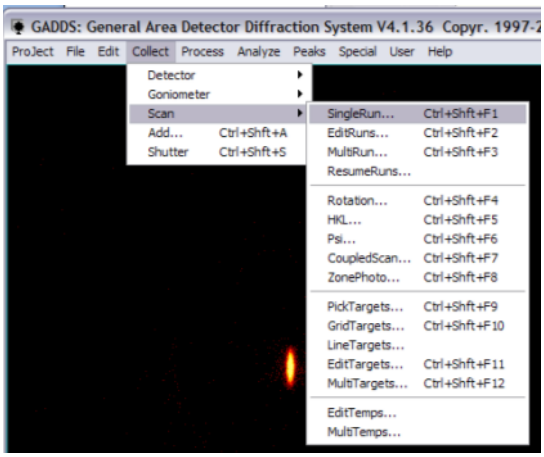
a. If you have already not done so, Create a new project folder (→ Project, →new, input information accordingly. Use the working directory C:\Frames\"year"\"month"\"Sample Name" (This automatically saves subsequent data collections to the above file path))
b. Make sure that the X-ray generator is at the operating power (→Collect, →Goniometer, →Generator)



Set to 40kV and 40 mA



c. Set up a single scan (→ Collect, →Scan, →Singlerun)



d. Set up for the data that you want to collect:
   i. Active field descriptions:
      - **# Frames**: How many frames of diffraction will be collected
      - **Seconds/Frame**: How long the detector will be exposed for each frame
      - **Scan Axis #**: This is which position/drive that will change between subsequent frames
        - The value "**Coupled**" requires both θ_1 and θ_2 to be the same value and they will move equally to create the desired 2θ value.
      - **Frame width**: How much the scan axis will change between each frame.
      - **Mode**: Usually "**STEP**"
      - **Rotate Sample**: This will control whether or not the sample will rotate about Phi during data collection
        - This is helpful if you have a highly textured sample or one with large grains
      - **Sample Osc**: What position (s), if any, will oscillate during data collection
        - This is useful for collecting x-ray data over a larger area of the sample to improve particle statistics for samples with larger grains
      - **Amplitude**: How much the axis will oscillate during data collection
        - The amplitude value is the distance from a zero position (i.e. an amplitude value of 0.5 would oscillate the sample 1 mm due to the ±0.5mm amplitude))
      - **Frame Header Information**
        - This is not necessary when creating a file name (it is only viewed in GADDS)
        - Misc. information that will be attached to your data for record keeping purposes
      - **Filename generation**: "Job name"_"Run #"_"Frame #"
        - Used to auto generate a filename
        - Change as needed (the "grayed-out" File name is how it will appear in the project folder)
      - **Max display counts**: This controls the intensity scale during the real time display of data
      - □ **Realtime Display**: This shows how the x-rays are being collected in real time
      - □ **Pre-clear**: This provides a clear screen at the beginning of each frame.
      - □ **Capture video image**: This will attach an image to your data files (as seen in the Laser-Video system)
      - □ **Auto Z align**: Never check this option. This finds the correct z-height of your sample automatically (it is better to do this manually, as in the previous steps)

   ii. Typical **SingleRun** setting for a new user is shown below (this will collect data from about $15° \leq 2\theta \leq 105°$)

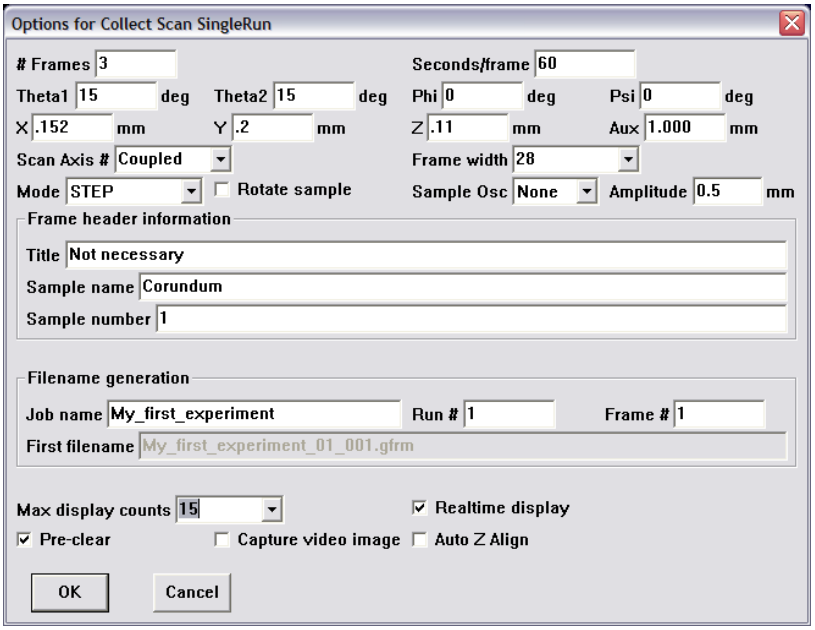
NOTE: When setting up a scan to collect data, be sure to consider where you expect diffraction peaks to appear (as in the PDF). It is not good practice to integrate the same peak in two different scans.

- **# Frames**: 3
- **Seconds/Frame**: 60-300

1. Θ_1: 15°
2. Θ_2: 15°
3. Phi: Use "**@**" (this tells the program to display the current axis value that was predetermined with the **Laser-Video** system)
4. Psi: 0
5. X: Use "**@**"
6. Y: Use "**@**"

7. Z: Use "**@**"
8. Aux: Use "**@**" (this is the camera zoom)

- **Scan Axis #**: Coupled
- **Frame width**: 20-30 (depending on the amount of overlap desired on integration)
- **Mode**: STEP
- □ **Rotate Sample**: Typically not used for powder diffraction
- **Sample Osc**: None
- **Amplitude**: 0.00 (This is only relevant if you are oscillating your sample)
- **Frame Header Information:** Change as necessary (only seen in GADDS)
- **Filename generation**: Specific to the sample and project
- **Max display counts**: 15
- ☑ **Realtime Display**
- ☑ **Pre-clear**
- □ **Capture video image**
- □ **Auto Z align**



e. Once the parameters are setup correctly, press the OK button to start the scan.
f. The data will be automatically saved as a .GFRM file in the project folder that was created in an earlier step.
g. The last frame collected will be shown in GADDS when the measurement is finished.
    i. To navigate to different Frames with in a Run, press "CTRL" + "→" or "CTRL" + "←"
h. Integrate the data create a one-dimensional scan for evaluation in **EVA**:
    i. Go to the frame of interest, then:
        1. →Peak, →Integrate, →Chi, →enter
        2. Select the range you are interested in
        3. Save the file in the folder of interest for further analysis using EVA. →Peak, →Integrate, →Chi, click OK
        4. Use 1, 2, 3, 4 to change the 2θ range and Chi range for integration
            a. If you are planning to merge multiple frames of different 2θ ranges, it is important to note the beginning and ending values of 2θ.
        5. Integrate the next frame in the series, → "CTRL" + "→" or "←"
            a. Then repeat the above steps
            b. Make sure you have at least 0.1 degree of overlap on the beginning and ending 2θ values, otherwise your scans will not merge together in EVA.

8. Remove sample.
    a. Take your sample out of the XRD (the BSCMC is not responsible for lost and/or damaged samples)

9. Close the GADDS operating software and leave the unit in standby mode (20kV, 5mA)
    a. → Project, →Exit

10. Update the iLab Calendar if necessary