

# Operating Procedure for Bruker D8 Discover X-ray Diffractometer

## Preparation:

- Log into login computer
- Check System Control Buttons (located at the lower right or left of the system. [see below]). Make sure the “Ready” and “On” LED lights are on. If the D8 is at low current and voltage (standby mode), the “Alarm” LED will blink because the chilled water is too cold. As the current/voltage is increased to maximum intensity (normal usage), the “Alarm” LED will stop blinking.



### Stop Button: Emergency Stop!

If hit, immediately switches off the control electronics and high-voltage generator. X-ray source is turned off and all moving drives will stop instantly

**Turn Off Power:** Switches off the control electronics, high voltage generator and all components connected to the AC outlets

**Turn On Power:** Switches on the control electronics, high voltage generator and all components connected to the AC outlets

**High Voltage Rotary Switch:** Turning the switch to the right position and hold until the orange “X-RAY ON” status LED starts flashing. Powers up High Voltage: “X-RAY ON” will turn solid orange when done powering up.

**Open Door Button:** Under normal operating conditions the door handles are locked by a mechanical latch. To open the front door(s) this button must be pressed first (to release the latch). Then the front door(s) can be opened. Pressing the “Open Door” button while the X-ray tube shutter is open will cause the shutter to close automatically.

## Before Loading Specimen:

- Before opening the D8 door, check for abnormalities in and around the instrument. On the back wall of the D8, the “X-RAY-ON” four orange LEDs and “SHUTTER CLOSED” four green LEDs lights should be lit.
- If **XRD Commander** is not opened, double-click the icon to open it. Once the software is opened, the computer will communicate with the D8 hardware.
  - If “Actual” position fields for the drives show dashes, such as --> the drives must be initialized. Select drives using the check boxes (preferably two at a time rather than all at once) and click “Init Drives”:



Theta 22.----



- Set the primary and secondary optics using pull-down menus: 
- There is only one “Default” primary.
- If the D8 setup is as shown on the reference page (end of document), the secondary optics is “Pathfinder”, with the path determined by your desired experiment (either Variable Slit or Triple Mirror). “Default” secondary corresponds to Soller slit in front of the detector mount. Use the table on the next page as a reference:

<u>Secondary Optic Path Name</u>	<u>Mounting location</u>	<u>Description</u>
"Default Optic"	front of detector at 300 mm. Soller slit in front	Used for grazing incidence detection (GID) or to maximize the amount of signal that reaches the detector
"Pathfinder: Variable Slit"	front of Pathfinder at 350 mm. Detector mounted at a diagonal in the back	Standard Bragg-Brentano measurements
"Pathfinder: Ge 220 Mirror"	front of Pathfinder at 350 mm. Detector mounted at a diagonal in the back	Mirrors cut down on signal. Very high resolution achievable.

### Start-Up:

- Increase voltage (kV) and current (mA) from 20/5 (standby) => 40/40 (operating)
- Set absorber to the maximum "5145" value to prevent saturation of the detector.
  - NOTE: after changing the pull-down, you have to click "Set" to the left of the menu for the change to occur.
- Move drives. Enter values into fields, mark the check boxes next to them and press "Move Drives" button (not to be confused with "Init Drives"). ----->  Also check "Aux" drives. If the values are different from the ones below, move them too.

<input type="checkbox"/> Std	Theta: 0	X: 0	<input type="checkbox"/> Aux	Alphal: 0
	2 Theta: 0	Y: 0		Zoom: 0.9999
	Chi: 0	<b>Z: -0.9</b>		
	Phi: 0			

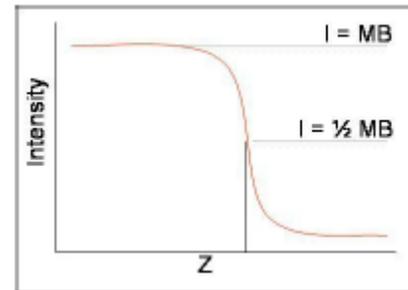
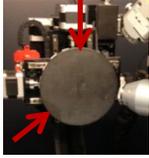
- Perform a **Detector scan** (-1 to 1). Select it in "Scantype" menu at the bottom of the window. Press "Start". If a peak is visible, zero initialize (ZI) the peak value. If there is no observable peak, confirm that you have selected the appropriate primary and secondary optics from the pull-down menu and try again. 
  - DO NOT click "ZI" if you do not see an observable peak as the software will try to find a peak amongst the noise and may significantly alter the zero value.
- Set absorber back to "Auto" value.

### Loading Specimen:

- To open the doors, press "Open Door" green button (on one of the system control panels) to disable the mechanical latch. Pull handles toward you and slide both doors open.
- Mount your sample at the center of the stage (large grey circle) using double-sided Scotch tape.
- To close the doors, slide the doors closed and push handles toward the back of the machine until you hear the "click" of the mechanical latch engaging.
  - **NOTE: If both handles are not firmly locked, DO NOT continue. Instead, press the "Open Door" button, open the door and shut it again. Failure to close the door properly could result in a full system lock.**

### Optimizing sample position in the beam path:

- Perform a **Z** scan (-0.9 to 1.9), setting the Z position center to the location correlated with intensity (I) at  $\frac{1}{2}$  maximum beam (MB)
  - If  $\frac{1}{2}$  MB is not visible, spacers need to be added or removed to move the sample to an appropriate z position.
  - Stage can be removed using two hex-key set screws located as shown to the left.
  - Spacers are metal plates with holes mounted beneath the stage. Each spacer thickness is twice the smaller-size. Default for thin film/materials is two spacers (smallest and medium size).
  - Double click on the curve where the intensity is  $\frac{1}{2}$  MB
- Perform a **Rocking Curve** (-1 to 1), double click on the peak location
  - NOTE: If the peak is not obvious on your sample, you are better off not double-clicking (leaving theta = 0). Any deviation from zero here will shift your 2Theta/Omega scan values later.
- Perform a **Z** scan (-1 to 1) again, double click on the curve where the intensity is  $\frac{1}{2}$  MB



The next steps are performed only for samples with a strong diffraction peak, position of which is known in advance (for example, single-crystal Si(111) shows a peak at 28.443°):

- Perform a preliminary **2Theta/Omega** scan in the range you expect a strong peak. Double click on the peak (this will enter the 2Theta value into the 2Theta field).
- Perform a **Rocking Curve** (+/- a few degrees equal to  $\frac{1}{2}$  the peak's 2Theta value). Double click on the peak (this will enter the Theta value into the Theta field).
- Perform a **Chi** scan (-3 to 3), double click on the peak if there is one.

### Data Collection:

- Perform a **2Theta/Omega** scan (note: "omega" = "theta" except that theta and 2 theta are uncoupled). Choose a 2Theta range that will encompass your target peak(s) and step size appropriate for the resolution you desire.
  - For optimal identification, make sure each peak has a minimum of 3 data points on the rising side and 3 on the falling side.
- Save the measured data as a ".RAW" data file by selecting "Save As" in File menu. You need to do it after each scan. Once you start a new scan, previously collected data is lost.

### Shut Down:

- Set voltage (kV) and current (mA) to 20/5 (standby mode).
- Minimize XRD Commander. **Do not close the program.**
- Remove sample(s) and clean up.
- Transfer data if necessary. Do not use USB storage devices on the machine's computer. Use network N: drive to copy data and pick up the files at the computer room ACEE 027D or send the files to yourselves through the internet.

### Laser pointer on/off

The red laser pointer marks the approximate location where the X-ray beam hits the sample. It may be useful for sample mapping. The laser can be turned on and off using “Direct Command”:

- Press “Direct Mode” button in the top menu
- To turn the laser on, enter “OC16, 1” in “Command” field, press “Send Command”
- To turn the laser off, enter “OC16, 0” in “Command” field, press “Send Command”

### Data Processing:

You can perform simple data processing in Eva software package

- Load the saved .raw file in **Eva**.
- Process the XRD result with options such as background removal, peak smoothing, and Y-scale etc.
- Export the processed result to another .raw file. (File > Export > Current Scan (whole data range))

**PDXL2** is an advanced XRD analysis software installed on Rigaku Miniflex XRD computer. It has an associated database that may be useful for phase identification in your spectra. PDXL2 accepts “.raw” file format. The software user guide is a part of Rigaku Instructions booklet.

- To transfer data files from Bruker XRD to Rigaku XRD computer you may use IAC Data Server. Copy your file to N: “IAC Data Server” network drive.
- Go to the Computer Room (ACEE 027D). Use one of the computers on the right side of the room. Open “IAC Data Server” drive, you will see folders from different IAC machines. Copy your data from “Bruker-XRD” to “Rigaku-XRD” sub-folder.
- Access your data on the “IAC Data Server” drive on Rigaku computer (You will need to login as a user to access this computer).

### Converting .raw to .uxd format (Excel/Origin Readable Format):

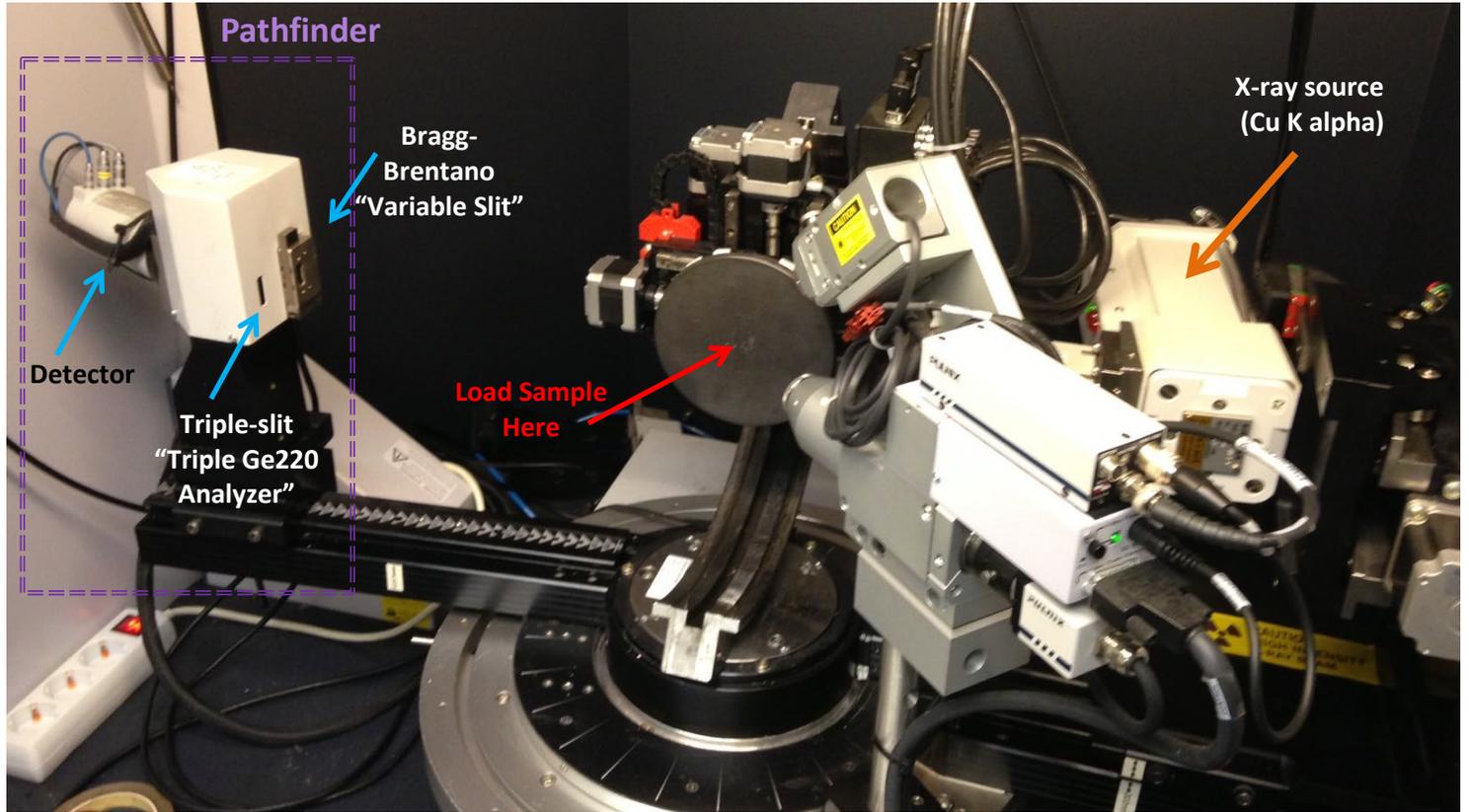
- Load the processed .raw file in **File Exchange**.
- Format the conversion parameter. (File > UXD Format).  
Raw Data Format: “Angle + Intensity”  
Peak List Format: “Angle + Intensity”  
Special Formatting: “Skip Headers Information” and “,” as the field separator.
- Convert the .raw to .uxd. (File > Translate > UXD).
- UXD can be opened by text editing software such as Notepad, Excel, and Origin etc.

### **Remember:**

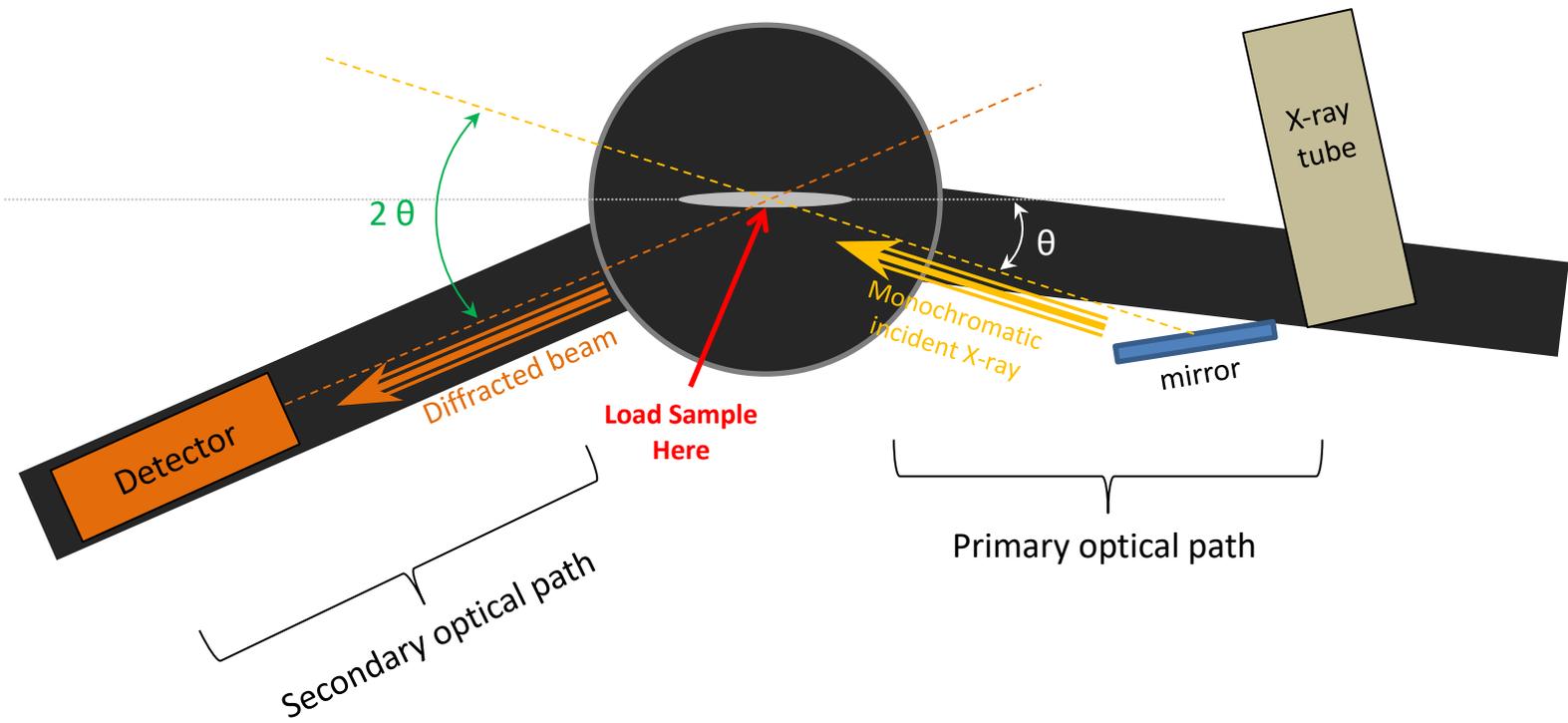
**Sign the logbook and LOGOUT from the Login system computer.**

\* Please contact Paul Shao (8-3851, [pshao@princeton.edu](mailto:pshao@princeton.edu), ACEE) or Nan Yao (8-6394, [nyao@princeton.edu](mailto:nyao@princeton.edu), 033 ACEE) if you have any problems.

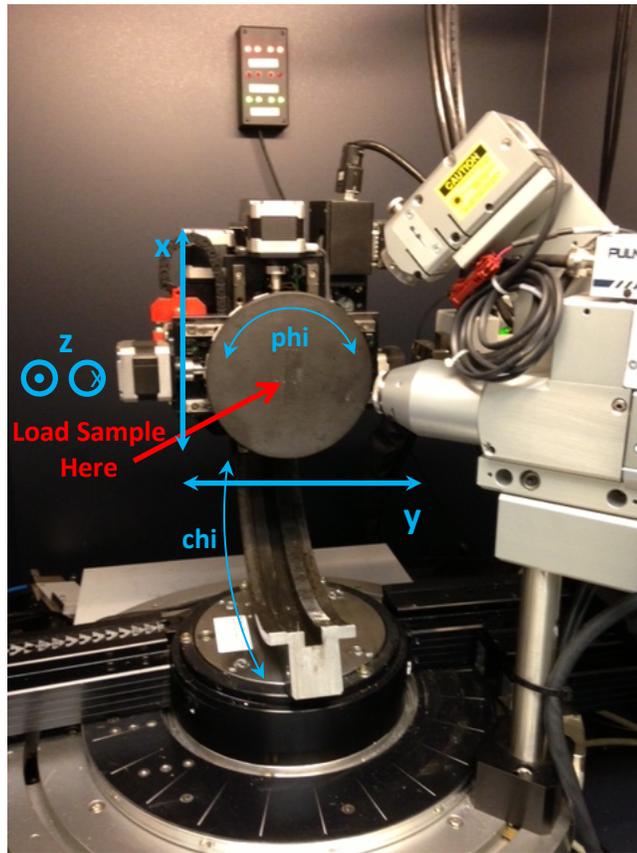
Reference Diagrams:



D8 Discover (Diagram)  
Top-Down View



Stage:



### **Emergency Information:**

Medical Emergencies: Contact 911 and Public Safety (609) 258-1000

Room / facility emergencies: Contact Public Safety (609) 258-1000

Issues related to the instrument:

1. Contact IAC Staff.
2. If unsure, leave system as is.
3. Try to turn off X-rays and shut down system.

Audible/Siren Emergency Alerts:

Follow previous steps 2 & 3 and leave the building.

### **Emergency Contact Information:**

Nan Yao: Office (609)258-6394; Cell (908) 922-2236 Email: [nyao@princeton.edu](mailto:nyao@princeton.edu)

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