**D2 Phaser XRD general SOP**

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For more information on the XRD instruments please visit http://argenta2.rd.unr.edu/x-ray-powder-diffractometer.html

**Before you get started please sign up for instrument time at http://argenta2.rd.unr.edu/mrbs**

**Log in to the D2 PC**

1. Log in with username “Stephen” and your password
2. Launch DIFFRACT.measurement from the Desktop shortcut (no password needed)

**Preparing the D2 for scan**

1. Prepare your sample in one of the round Bruker sample holders
2. Samples should be prepared in your lab **not the XRD lab**
3. See D2 Bruker videos on the argenta2 server for sample preparation
4. Make sure the X-rays are off (LED row on top of instrument is off)
5. If you accidently push up on the front door while the x-rays are on it will not open very far; push down on the black bar to fully close it before proceeding, otherwise the safety circuit is triggered and you will not be able to run a scan
6. Load your sample into the D2
7. Open the front door by pulling on the black latch behind the front handle
8. Lower the rotating stage by pulling down the black knob inside the instrument
9. Insert your sample on the rotating stage
10. Raise the rotating stage by pushing the black knob all the way up until you feel it click into place
11. Select the desired air scatter height: 1mm for low angles, 3mm for general use
12. Make sure the needed slits are in; for general use insert 0.6 mm slit on the primary side and no secondary air scatter slit on the secondary side; if you change these hardware settings, please make a note on the daily log sheet!
13. Close the front door of the D2 Phaser by pulling the front handle on the door down until you hear it click into place

4) Initialize drives:

1. In “Commander” in the top left panel, anything with a caution symbol needs to be initialized. Click the checkbox to the right of each drive value and then click the “Initialized all checked drives” button, agree “OK”

***The system is ready for scanning***

**Scan settings for D2 and managing your results**

***NOTE:*** *The numbers given below are just standard settings and you are free to change them to what you need.*

*The numbers that you should* ***NEVER*** *change are the x-ray VOLTAGE and CURRENT as well as the STEPS parameter.*

1. Choose scan parameters and start scan:
2. If not done already Initialize drives (see above)
3. Start sample rotation manually before starting the scan by typing in the desired speed (standard is 15 rpm) and hitting the “Apply new values” button right next to it; Phi should start to rotate
4. In the “Scan setup” panel fill in desired scan settings (see below for general settings); click into a white box several times to update the Effective total time [sec]; adjust dwell time and increments if total time does not fit your needs
5. In the “Generator setup” panel fill in parameters for x-rays (30 KV, 10 mA = 300W), then hit ON to turn them on (SET will not work in this case); the top of the D2 instrument should light up with a row of LEDs
6. Hit START; the drives go to the starting position and the scan is started
7. **After each scan, save your results file or the data will be lost!** When the scan has finished go to FILE > SAVE RESULTS ***both*** as .brml and .raw in a local folder and only then prepare next scan or sample
8. Before you change samples turn off X-rays in the “Generator setup” panel, make sure LED’s on top of the instrument are off

**General Settings for Data Acquisition:**

Sample rotation: 15 rpm

2Theta angles: Your choice; depends on your sample type

Time (s): Your choice; more time equals better signal/noise (S/N)

Increment (°): Your choice; a smaller increment equals better S/N and you should set the increments so you get at least 5 data point above the half-height

Scan type: Coupled two theta/theta

Scan mode: Continuous PSD fast (Position Sensitive Detector)

PSD opening: 4.84876814600

***NOTE:*** *Do* ***NOT*** *adjust the STEPS parameter, it is automatically calculated from the other parameters. By adjusting steps improperly you can damage the optics!*

**Ending your session**

***NOTE:*** *The .brml and .raw files that you have saved so far can only be opened with DIFFRAC.EVA and JADE; please follow the steps below to make your data accessible to you*

1. Data processing:
2. Turn off X-rays in the “Generator setup” panel and close DIFFRACT.measurement; the LED’s on top of the instrument should be off by now
3. Import your .brml files in DIFFRAC.EVA and then export them as .xy file; this file extension can be opened in notepad, it will have two columns, 2Theta and CPS, which can be copied into excel.
4. In DIFFRAC.EVA you can also search/match and append the matches if you like, and then print your spectrum.
5. You can install DIFFRAC.EVA on your windows PC, just follow the instructions on the argenta2 XRD webpage (see link on top of first page); note that this software can only be used within the chemistry domain network
6. If you want to refine your scans in Jade please ask the SIL staff to arrange Jade usage. The .raw file will be needed for that purpose.
7. Copy saved files into your argenta2 folder (file browser Y: data \\argenta2.rd.unr.edu > Bruker D2) or on a thumb drive
8. Log off from Windows

***Keep D2 and the PC ON except for maintenance***

**Turning on the D2**

***NOTE:*** *The D2 should be turned on already as it only needs to be shut down for maintenance; if it is not up and running please let the SIL staff know*

1. Turn on the power to the D2, the switch is located at the back of the instrument on the lower left-hand side
2. Wait for the embedded Windows interface to fully load (you should see a desktop with the time on it and a Bruker logo)
3. Start the external Windows PC
4. Log in with username “Stephen” and your password
5. Launch DIFFRACT.measurement from the Desktop shortcut (no password needed)

**Shutting down the D2**

**NOTE: The D2 only needs to be shut down for maintenance reasons**

1. Shut down the computer:
2. In DIFFRACT.measurement turn off the X-Ray generator
3. Exit DIFFRACT.measurement (some settings in “Commander” tab will be lost) and DIFFRACT.eva
4. Shut down computer
5. Shut down the embedded Windows on the D2 Phaser:
6. Push on the bottom drawer on the D2 to release the keyboard and mouse drawer, use the mouse to click on screen
7. On the lower right-hand side of the screen left-click on the power icon and select shutdown
8. When you get the message “Windows was shut down successfully. It is now safe to switch off the instrument” proceed to step 3
9. Power off the instrument using the switch on the back of the instrument on the lower left-hand side

***Keep UPS ON except for UPS maintenance***