**D8 Advance XRD with Rotating stage SOP**

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

**Turning on the D8 (with desired stage already attached)**

1. Start the cooling cycle:
2. Open up the two water mains valves on the wall between the desks (handles have to line up with the lines)
3. Switch on the Haskris chiller behind the D8 PC
4. Turn on the instrument:
5. On the lower left-hand side of the instrument, turn the large yellow-and-red Mains knob from O to I, wait 15 seconds
6. Press the green “Power-on” button next to the Mains knob
7. Wait for the “I” to appear on the top button on the left-hand front of the instrument (generator button, takes roughly 50 seconds)
8. Once “I” has appeared press that same generator button; it will start blinking yellow and after 3-4 seconds it should become a black on yellow radiation symbol; when cathode is fully heated up the symbol will turn to yellow on black

***NOTE****: After 2 days powered down then the instrument will do a 45 minute tube conditioning (generator button flashing blue with “COND” on it). When the instrument is finished doing the tube conditioning, the generator button will become a yellow on black radiation symbol*

1. Start the computer:
2. Turn on the computer – ask Nina or Stephen for password
3. Launch DIFFRAC.measurement from bottom task bar, log in using Bruker Service with password “Bruker-Service”
4. After a while you should see the Measurement Server starting. Open it (task bar, right side), hit “Connect”, then close window

***NOTE:*** *If this is the first launch after everything has been off it will take a while for everything to connect*

**Preparing the D8 for scan with rotating stage**

1. Prepare sample in the rotating stage sample holder:
2. To open the door to the XRD push the door open button ->||<- on front right-hand side of instrument; even if the x-rays are on the shutter will shield them from exiting the primary optics (check the LEDs on the x-ray tube for shutter status, if they are bright red the shutter is open)
3. The rotation stage has a sample holder that is magnetically attached, simply pull down to remove it
4. Load/unload samples in the magnetic metal holder ***OUTSIDE*** of the XRD cabinet. The holder is spring loaded and we do not want any sample spill inside the XRD cabinet. Make sure the sample surface is flush with the surface of the holder, otherwise your peaks will be shifted
5. Re-insert the rotating stage sample holder slowly but with a firm grip, it will snap in place and if you let it snap without a firm grip your powder sample could move around
6. Adjust the knife air scatter screen positioned above the sample surface to properly remove background air scattering. Using the thumbwheel near the top, move blade up/down according to incident angles used (3mm above sample surface is standard; lower it for very low angle scans)
7. Choose primary (X-ray tube) and secondary (detector) optics in “Commander”:
8. In DIFFRAC.measurement, go to the “Commander” tab and wait until everything is connected
9. Setup if using the **motorized slits**:

* Twin Primary: Motorized Slit: Slit width 0.681. You must hit the “Apply new values” button to activate the setting
* Twin Secondary: Motorized Slit: Slit width 0.681. You must hit the “Apply new values” button to activate the setting
* **Insert** **the Nickel** filter on the secondary side of the optics

1. Setup if using the **Göbel mirrors**:

* Twin Primary: The Göbel mirror is more forgiving of poorly ground/defined samples (seems to give better S/N)
* Twin Secondary: Motorized Slit: Slit width wide open at 5.4 mm. You must hit the “Apply to settings” button to activate the setting
* **Insert a** **fixed slit** (standard is 0.1 mm) on the primary side or your peaks will be broad, and **remove the filter** on the secondary side since the radiation is already removed by the Göbel mirror

4) Initialize drives and x-ray tube:

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 “yes”
2. In the lower X-ray generator section, set Voltage to 40 kV and Current to 40 mA, hit the SET button

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

***NOTE:*** *After 5 min in idle the system will ramp down the Voltage and Current to 20 kV and 5 mA*

**Scan settings for D8 and managing your results**

***NOTE:*** *The numbers given below are just standard settings and you’re free to change them to what you need. The numbers that you should* ***NOT*** *mess with are the x-ray power and current as well as the STEPS parameter.*

1. Choose scan parameters and start scan:
2. If not done already, Initialize drives and x-ray tube (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. Hit START; the X-rays will turn on, power is ramped up, drives go to starting position and the scan is started
6. **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

**General Settings for Data Acquisition:**

Sample rotation: 15 rpm

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

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

Increment (°): Your choice; a smaller increment equals better S/N but 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 crash the optics!*

**Ending your session and shutting down the D8**

***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. Close DIFFRACT.measurement
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 to 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 Nina or Stephen 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
9. Turn off the instrument:
10. Press the generator button on the top left-hand front of the instrument (after a few seconds the button icon will change from a radiation symbol to a white “I”)
11. Press the white “Stand by” button in the insert on the lower left hand side of the instrument
12. Turn the big yellow-red Mains knob from I to O (next to the Stand by button)
13. Stop the cooling cycle:
14. Switch off the Haskris chiller
15. Close water mains (2 valves) on wall; handles should be perpendicular to lines

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

**Removing the Rotating Stage**

1. Before switching stages make sure the goniometer arms are out of the way by reinitializing the drives (Commander, top checkbox, Initialized all checked drives)
2. Shut down the computer:
3. Exit DIFFRAC.measurement (some settings in “Commander” tab will be lost)
4. Open Measurement Server (task bar, right side), hit “Release control”, then close window
5. Shut down computer
6. Turn off the instrument:
7. Press the generator button on the top left-hand front of the instrument (after a few sec the button will change from radiation symbol to a white “I”)
8. Press the white “Stand by” button on the lower left hand side of instrument
9. Turn the Mains power knob from I to O (next to the stand by button)

***!!! NOTE: You must turn off the Mains power to the instrument before exchanging a stage, if not you will damage the driver board !!!***

1. Remove the rotating stage:
2. To open the door to the XRD push the door open button ->||<- on front right-hand side of instrument; even if the x-rays are on the shutter will shield them from exiting the primary optics (check the LEDs on the x-ray tube for shutter status, if they are bright red the shutter is open)
3. Remove the knife edge (air scatter) and place the sheath on
4. The rotation stage has a sample holder that is magnetically attached, just pull down to remove it
5. Using a T25 wrench remove the outer 3 retaining screws on the goniometer (twelve o’clock, four 0’oclock, and eight o’clock positions): ***note*** the screw at twelve o’clock is longer than the other two. Place the screws in the bottled labeled “***Rotating Stage Screws***”
6. ***NOTE:*** If attaching the rotation stage you will have to tighten the centering screw (on the goniometer flange to the left of the center) before tightening the three screws, and then release the centering screw again
7. Turn the whole stage counter-clockwise 45 degrees and pull straight out
8. Disconnect the driver cable (beige cable head) on the back of the rotating stage using a small flathead screwdriver and then place the rotating stage somewhere inside the instrument but out of the way (I used left side)
9. The driver cable for the rotating stage needs to be removed from the center of the goniometer. Using your left hand feed the cable through the hole in the goniometer and give it to your right hand then move the cable to the front of the cabinet

***The rotating stage is now considered completely removed from the instrument. The status of the stage can be viewed in DIFFRAC.measurement > DAVINCI tab***