**D8 Advance XRD with MTC-LOWTEMP Temperature chamber SOP**

5/21/2018 Lab manager: Janina Ruprecht jruprecht@unr.edu Cell 917 476 5641

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

**Temperature range: -180° to +450° C**

For temperatures < 25° C or temperatures > 300° C vacuum is required

For temperatures < 25° C cooling with LN2 is required

**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 temperature stage**

1. Prepare sample in Temp chamber 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. Vent the sample chamber by cracking open the large vacuum flange, then ***SLOWLY*** crack open the black vacuum break knob (back left hand side of stage; if you do this too quickly your sample will be blown throughout the entire sample chamber), wait until hissing subsides and vacuum flange is easily removed
4. Load/unload samples in the sample holder ***OUTSIDE*** of the XRD cabinet. We don’t want any sample spill inside the stage
5. Using the Wera 118064 micro screwdriver (black handle with green markings) loosen the thermocouple screw and remove the thermocouple
6. Loosen two screws at the bottom of the sample holder with a 2.5 mm wrench, pull out the sample holder while pulling down on the cold finger
7. Use a square of yellow post-it’s to stand the sample holder upright, add sample to the sample holder and level out with a glass slide
8. Place the sample holder back into the instrument stage by pulling down on the cold finger, tighten the two screws at the bottom of the sample holder with a 2.5 mm wrench
9. Reinsert and tightened the thermocouple using the Wera micro screwdriver
10. replace the vacuum flange and close it tight
11. Make sure the Haskris chiller is on, then turn on source and sink water valves for the stage, 2 yellow water valves on the floor behind the instrument. Make sure the pressure is set to 24 psi (pressure gauge on water hose going to instrument)
12. Turn on Eurotherm: Inside lower front console panel, press white button followed by green button; check in Commander if Temperature control is available in the panel below the X-ray generator panel
13. Set up vacuum (T < 25° C or > 300° C, or if desired) and cooling (T <25° C) if desired:
14. Close both black vacuum break knobs (back left and right hand side of the stage, both pointing sideways when closed)
15. Turn on vacuum pump
16. Turn on vacuum gauge
17. For cooling < 25° make sure there is at least 20 lbs of liquid nitrogen in the dewar
18. Turn on compressed air (up on the wall above the Haskris chiller)
19. Adjust compressed air pressure regulator to 0.5 bar

3) Choose primary (X-ray tube) and secondary (detector) optics in “Commander”:

1. In DIFFRAC.measurement, go to the “Commander” tab and wait until everything is connected
2. 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** $K\_{β} $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** $K\_{β}$**filter** on the secondary side since the $K\_{β}$ 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*

**Controlling the MTC-LOWTEMP Temperature Chamber (with everything ON)**

1. In the Commander tab you should see a Temperature control panel below the X-ray generator panel
2. Select whether you want just a straight temperature increase, or temperature with ramp (here you can adjust the ramping speed between 0-12 °C/min)
3. Type in your desired temperature and ramp speed
4. Hit the “Apply settings to instrument” button; the temperature should now start to ramp
5. When the stage has reached your desired temperature, wait until the temperature is stable (usually overshoots by 5° C), then run your XRD scans

**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. 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
4. Hit START; the X-rays will turn on, power is ramped up, drives go to starting position and the measurement is started
5. **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 temperature stage
10. Set the temperature to 25° C and wait until the system reaches 25° C
11. Once the system reaches 25° C turn off the heater on the Eurotherm (red button) then turn off the power to the Eurotherm (white button)
12. Close the source and sink water lines on the floor behind the instrument
13. Turn off vacuum pump (if used)
14. Turn off vacuum gauge (if used)
15. Turn of compressed air on the wall above the Haskris chiller (if used)

***The Temp stage is now fully turned off***

1. Turn off the D8 instrument:
2. 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”)
3. Press the white “Stand by” button in the insert on the lower left hand side of the instrument
4. Turn the big yellow-red Mains knob from I to O (next to the Stand by button)
5. Stop the cooling cycle:
6. Switch off the Haskris chiller
7. Close water mains (2 valves) on wall; handles should be perpendicular to lines

***Keep UPS PowerVar ON except for UPS maintenance*Installation of MTC-LOWTEMP Temperature Chamber**

Temperature range: -180° to 450° C

For temperatures < 25° C or temperatures > 300° C vacuum is required

For temperatures < 25° cooling with LN2 is required

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

***\*\*\* NOTE: you must turn off the mains power to the instrument before removing a stage, if not you will damage the driver board \*\*\****

1. Turn off the instrument:
2. 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”)
3. Press the white “Stand by” button on the lower left hand side of the instrument
4. Turn the Mains knob from I to O (next to the stand by button)
5. Remove the currently attached stage by following the instructions in the respective SOP
6. Attach the Temp stage:
7. Using a 4 mm wrench remove the screws from the storage stage
8. Lay the stage on its face and insert the high tension power cables in the cylindrical brass plugs, use a 2 mm wrench to loosen and tighten the terminals of the cables
9. Align the red dots on top of the goniometer and on the Temp stage goniometer flange, push goniometer flange in, turn whole flange clockwise 45 degrees
10. Connect the safety circuit cable (silver)
11. Connect the water lines (usually blue and yellow coming from the bottom of the instrument are connected together): white – white; yellow – yellow; blue – blue
12. Connect green thermocouple
13. Connect black line (vacuum sensor)
14. Tighten the single centering screw on the goniometer flange to the left of the center; this centers the stage to the goniometer
15. Using a T25 wrench replace the outer 3 retaining screws on the goniometer (twelve o’clock, four o’clock, and eight o’clock positions): ***note*** the screw at twelve o’clock is longer than the other two
16. Release the centering screw

***The Temp stage is now fully attached. The status of the stage can be viewed in DIFFRAC.measurement > DAVINCI tab***