XRD2 Operating Procedure – TTK 600 Stage

Temperature range: -150C to 450C in air (with LN2 cooling)
-190C to 600C in vacuum (with LN2 cooling)
See TTK 600 Guide

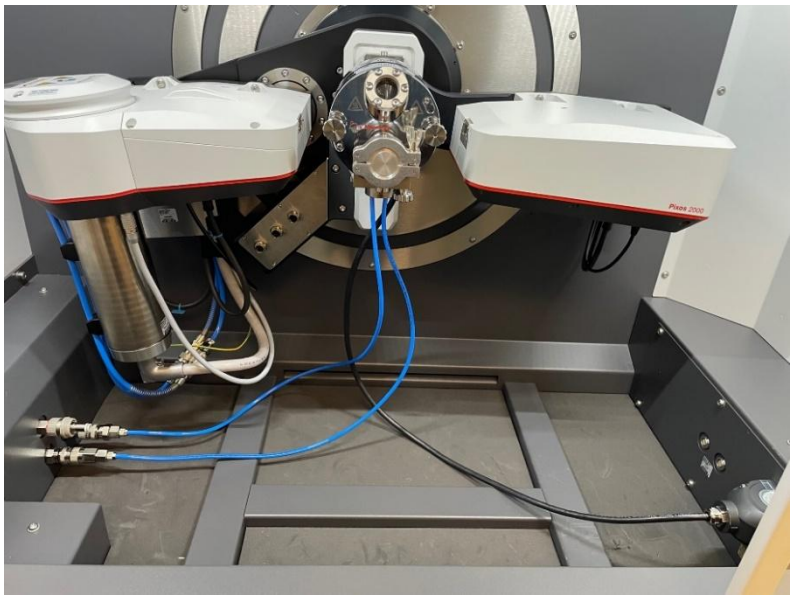
1. Ensure you have an XRD2 reservation in CoreResearch, and "Start" your reservation

Start-Up

2. Turn on power on front instrument panel
3. Wait until the power button stops flashing and all buttons on the front panel are illuminated (this takes about 1 minute)
4. Open XRDdrive software
5. Click the Connect button
6. Turn on X-Ray source
   - Wait until X-Rays have ramped up to 40kV and 49mA before proceeding

Experiment Setup

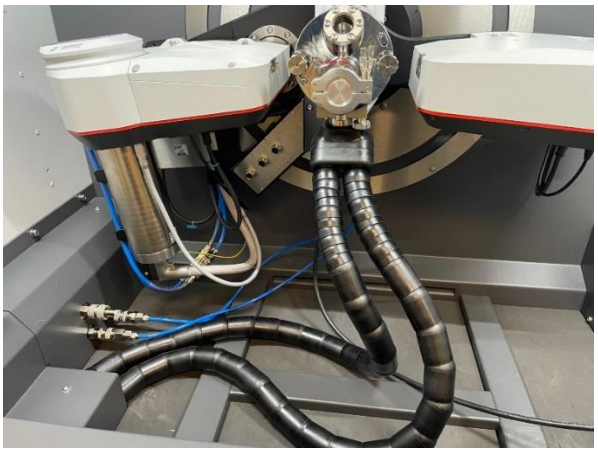
7. Press Door Open on the front instrument panel and then slide doors open
8. Unmount the Sample Spinner Stage (if in place)
9. Mount the TTK 600 stage and make all necessary connections
   - Connect the controller cable to the XRD chamber
   - Connect the cooling water lines



10. Load the sample into the TTK 600 stage *(see pages 27-29 in the TTK 600 guide*
11. If the experiment requires LN2 cooling (*see page 11 in the TTK 600 guide)*:
    - Fill the LN2 dewar (from the Sample Prep lab)
    - Connect LN2 hoses to bottom of TTK 600 and feed the free end of the hoses through the XRD cabinet opening

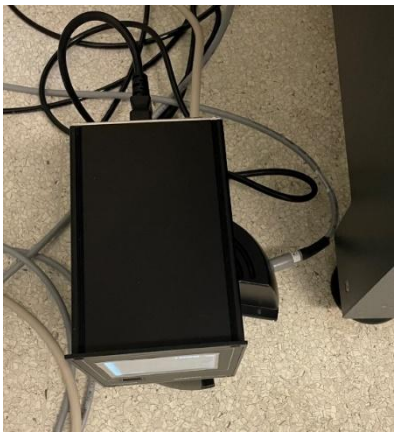
Note: If performing the experiment under vacuum, feed the vacuum pump cords through the XRD cabinet opening (step 12) prior to feeding the LN2 hoses through the XRD cabinet opening

- Connect the LN2 outlet hose (the long connector) to the Venturi-Nozzle safety box and the LN2 inlet hose (the short connector) to the LN2 dewar
- Connect the exhaust hose to the Venturi-Nozzle safety box and feed the free end through a hole in the opposite wall leading to the chase
- Connect the air line to the Venturi-Nozzle safety box and turn on the air supply on the back wall

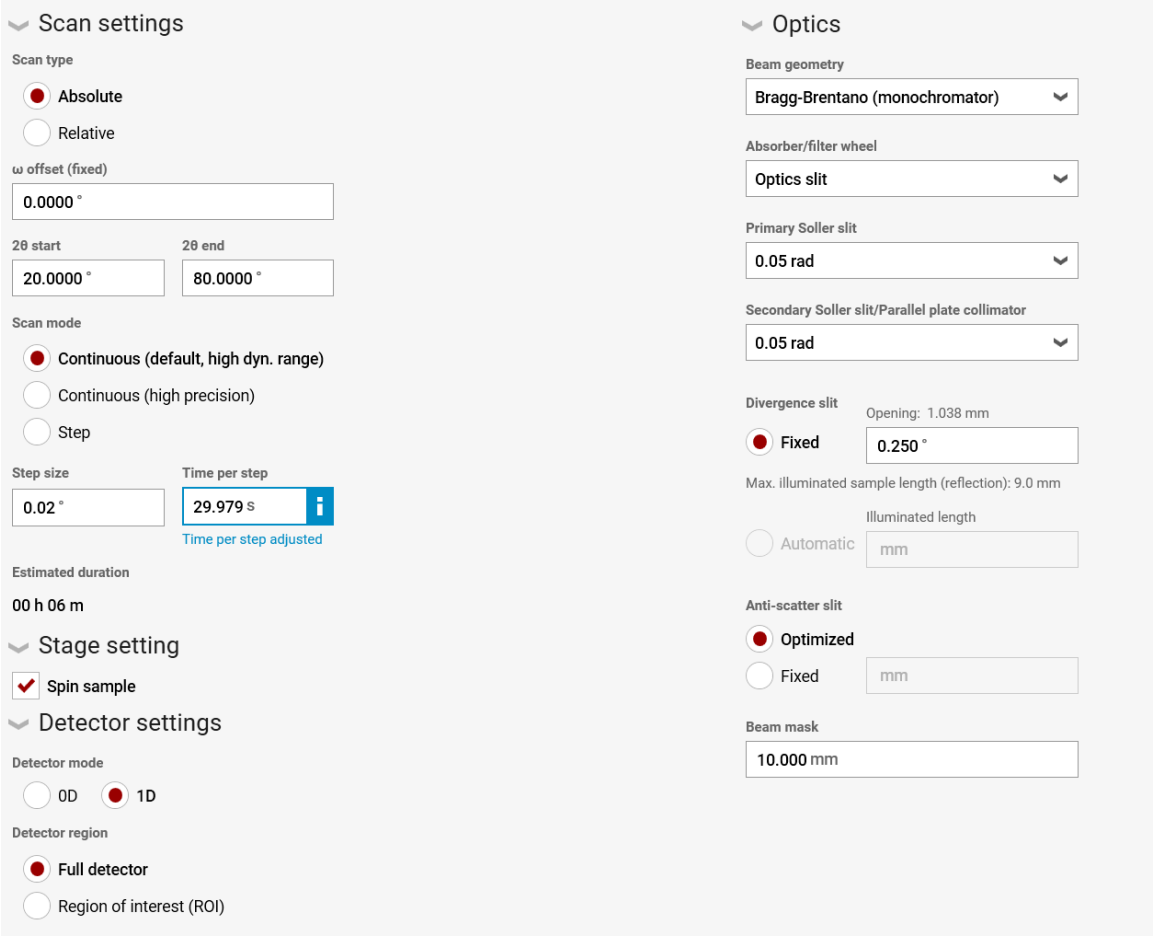


12. If the experiment is to be performed under vacuum:
    - Connect the turbo pump attachment to the front of the TTK 600 stage
    - Feed the turbo pump power cord and pressure gauge cord through the XRD cabinet opening and connect the vacuum hose
    - Connect the turbo pump power cord and pressure gauge cord
    - Turn on the pressure gauge and roughing pump
    - Turn on the turbo pump

- The pressure should get down to the low 10-4 mbar range



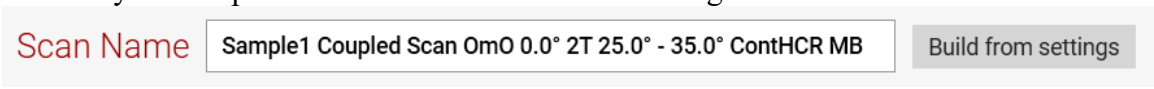
13. Close the XRD chamber doors
14. In the XRDdrive software, select either “Heating Only” if no LN2 cooling issued or “LN2 Cooling” if LN2 cooling is being used under the Select Stage Configuration pull down menu
15. Click Perform Automatic Stage Alignment and follow instructions
    - When completed, click close
16. Click on the menu icon and select either 
    - New Measurement Plan (to create a new measurement plan from scratch)
    - Open Measurement Plan (to open a saved measurement plan and edit)
17. Enter experiment name (the name of your measurement plan)
18. Select the Scan Data Target Folder by clicking on the icon function
    - Select or create your own folder in the XRDExperiments folder
19. Enter a sample name
20. Build your Measurement Plan
    - Select “Set Temperature” to enter a table of temperature values for your measurements
    - Select “Add Task” to insert a Wait step or Sample Alignment Step
    - Select “Add Scan” to create a measurement program
        - Use Coupled Scan for standard 2-theta measurements (moves both the incident X-Ray source and the detector)
        - Use Detector Scan for grazing incidence measurements (keeps incident X-Ray angle the same and only moves the detector)
        - Typical Settings are shown on the next page



Scan Type: Absolute
w offset: 0.0000
Scan Mode: Continuous
Step size: 0.01 or 0.02 degrees
Time per step: select so that total time is between 5-10 minutes
Spin sample: checked for powders, unchecked for films
Detector Mode: 1D
Detector Region: Full Detector
Beam Geometry: Bragg-Brentano (monochromator) or Bragg-Brentano (no optic)
Absorber/Filter Wheel: Optics Slit if using monochromator, Ni KB filter if no optic
Primary Soller Slit: 0.05 rad
Secondary Soller Slit: 0.05 rad
Divergence Slit: Fixed; type in a value such that the illuminated sample length is less than the actual sample length
Anti-Scatter Slit: Optimized
Beam Mask: type in a value that is less than the actual sample width

See "Influence of Selected Optics" document on SMIF XRD2 web page

- Click “Build from settings” to enter a scan name based on your setting values. Add your sample name to the front of this text string



Note; The raw data will be saved with a filename given by the scan name

21. Save Measurement Plan by clicking on the menu icon and choosing Save Measurement and then selecting your folder in the XRDExperiments folder

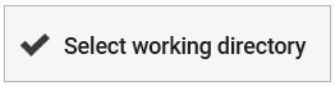
Experiment

22. Click Start Measurement to run your Measurement Plan
23. The data is automatically saved in two files:
    - .scn : text file of the raw data (can be opened in Notepad)
    - .hdf5: file containing the meta data (can be opened in HDFView)
    - A separate file is saved for each temperature step, and the temperature value will automatically be appended to the filename

Data Saving, Viewing, and Analysis

Data Viewing (*See XRDView guide)*

24. Open XRDview
25. Select “Start viewing data”
26. Click “Select working directory” and go to the directory that contains the scans you’d like to view.

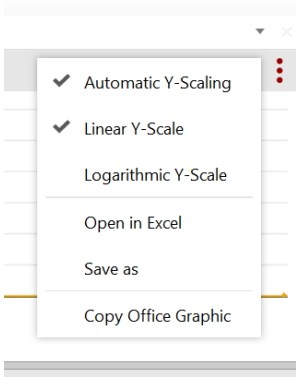


27. Selecting a scan will show the meta data for that scan in a side window
28. Select multiple scan by holding down Ctrl
29. Click the Select box in the bottom right corner to view the scans
    - If multiple scans have been selected, an overlay plot will be generated
    - Open the side window pane to adjust plotting plot settings
    - Plots can be saved in picture form by selecting “Export Chart”

Data Analysis (*See XRDanalysis guide)*

30. Open XRDanalysis
31. Create New Project
32. Add Measurement Data (select the hdf5 file you would like to analyze)

33. To save the raw data to Excel:
    - Click the 3 dots in the upper right corner and select “Open in Excel”
    - Save the Excel file to your folder in the XRDExperiments folder
34. Perform Data Analysis if desired
    - Search peaks
    - Fit peaks
    - Match (to files in ICCD database)
    - Quantitative Fit



Shut down

35. Press Door Open on the front instrument panel and then slide the doors open
36. If LN2 cooling was used:
    - Turn off the air supply on the back wall and disconnect the air line from the Venturi-Nozzle safety box
    - Disconnect the exhaust hose to the Venturi-Nozzle safety box
    - Disconnect the LN2 outlet hose (the long connector) from the Venturi-Nozzle safety box and the LN2 inlet hose (the short connector) from the LN2 dewar
    - Open the XRD chamber doors
    - Feed the free end of the LN2 hoses back up through the XRD cabinet opening, and then disconnect the LN2 hoses from the bottom of the TTK 600.
37. If the turbo pump assembly was used:
    - Turn off the turbo pump then turn off the rough pump
    - Turn off the pressure gauge
    - Vent the chamber by disconnecting the vacuum hose from the XRD chamber. Replace caps on connections.
    - Disconnect the turbo pump power cord and pressure gauge cord and feed them back up through the XRD chamber
    - Disconnect the turbo pump from the front of the TTK 600 stage. Replace caps on connections.
38. Wait until Sample Stage Temperature is less than 50C
39. Unload the sample from the TTK 600 stage
40. Disconnect the cooling water lines
41. Disconnect the controller cable from the XRD chamber
42. Unmount the TTK 600 stage
43. Remount the Sample Spinner Stage
44. Close the XRD chamber doors
45. In the XRDdrive software, a window should appear indicating that the Sample Spinner stage is mounted
    - Click on “Load Existing Stage Alignment”
46. Turn off X-Rays (if not already off)
47. Close Software
48. Turn off power on front panel
49. Stop your XRD2 reservation in CoreResearch