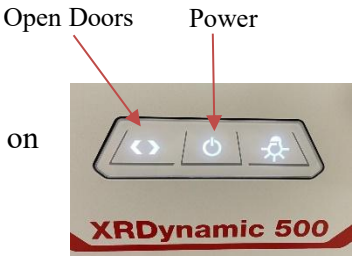
XRD2 Operating Procedure - HTK 1200N Stage

Temperature range: 25C to 1200C in air or vacuum

See HTK 1200N Guide

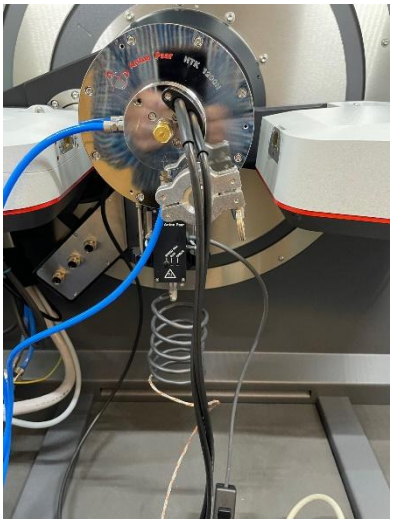
1. Ensure you have an XRD2 reservation in CoreResearch, and "Start" your reservation

Start-Up



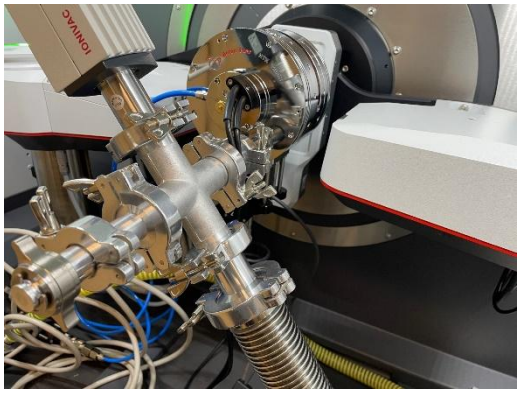
2. Turn on power on front instrument panel
3. Open XRDdrive software
4. Wait until the power button stops flashing and all buttons on the front panel are illuminated (this takes about 1 minute)
5. Click the Connect button
6. Turn on X-Ray source
    - Wait until X-Rays have ramped up to 40kV and 49mA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

Experiment Setup

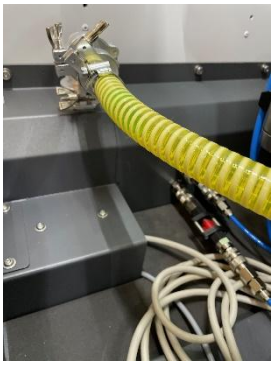


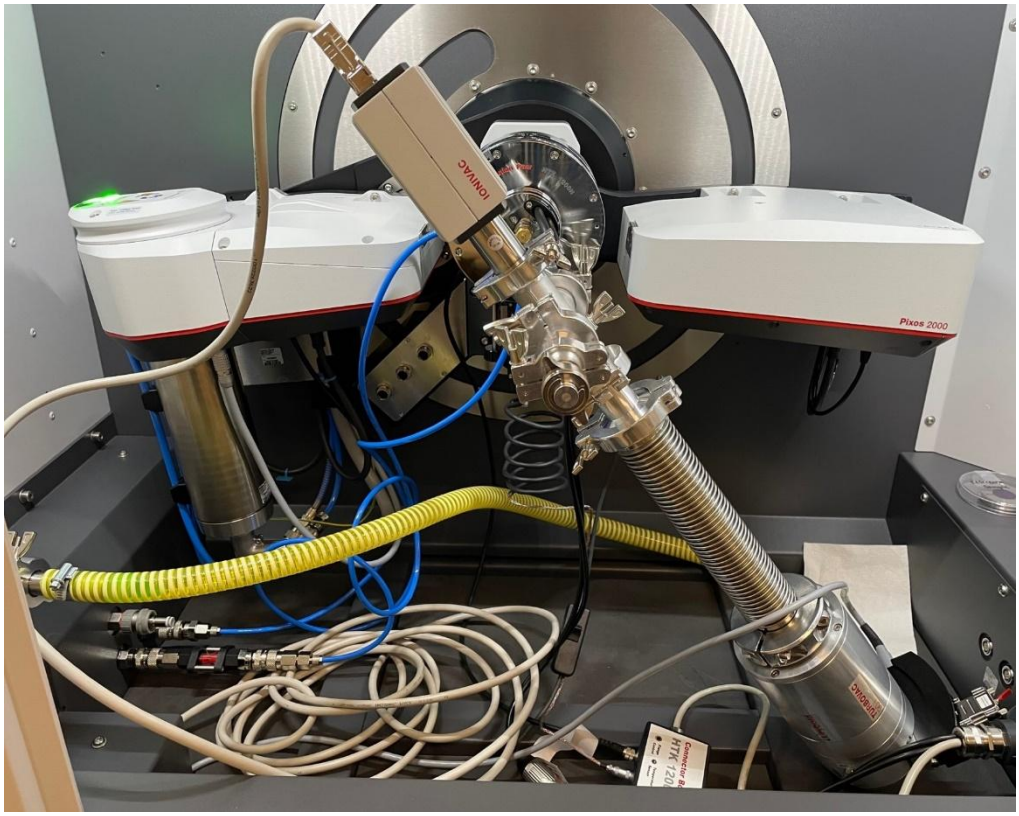
7. Press Door Open on the front instrument panel and then slide doors open
8. Unmount the Sample Spinner Stage (if in place)
9. Mount the HTK 1200N stage
10. Load the sample into the HTK 1200N stage *(see page 23 in the HTK 1200N guide)*
11. Make all necessary connections
    - Connect the heater power supply cable
    - Make connections to the HTK 1200N Connector Box
        - Connect the box to the XRD chamber
        - Connect spinner switch to the Connector Box
        - Connect flange control to the Connector Box
        - Connect one end of thermocouple to the Connector Box and the other end to the HTK 1200N stage. ***Handle carefully as the thermocouple wire and connections are fragile***
    - Connect the cooling water lines

12. If the experiment is to be performed under vacuum:
    - Connect the turbo pump attachment to the front of the HTK 1200N stage using adapter from the smaller diameter port of the stage to the larger diameter port of the turbo pump attachment

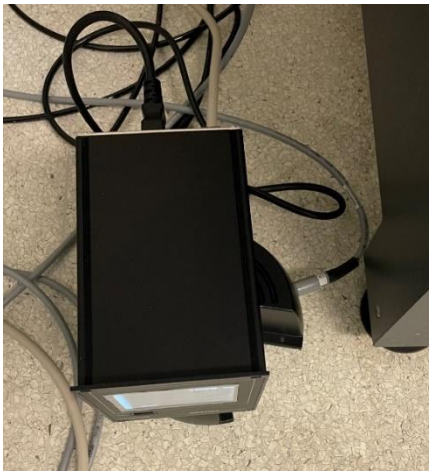


    - Feed the turbo pump power cord and pressure gauge cord through XRD cabinet opening and connect vacuum hose



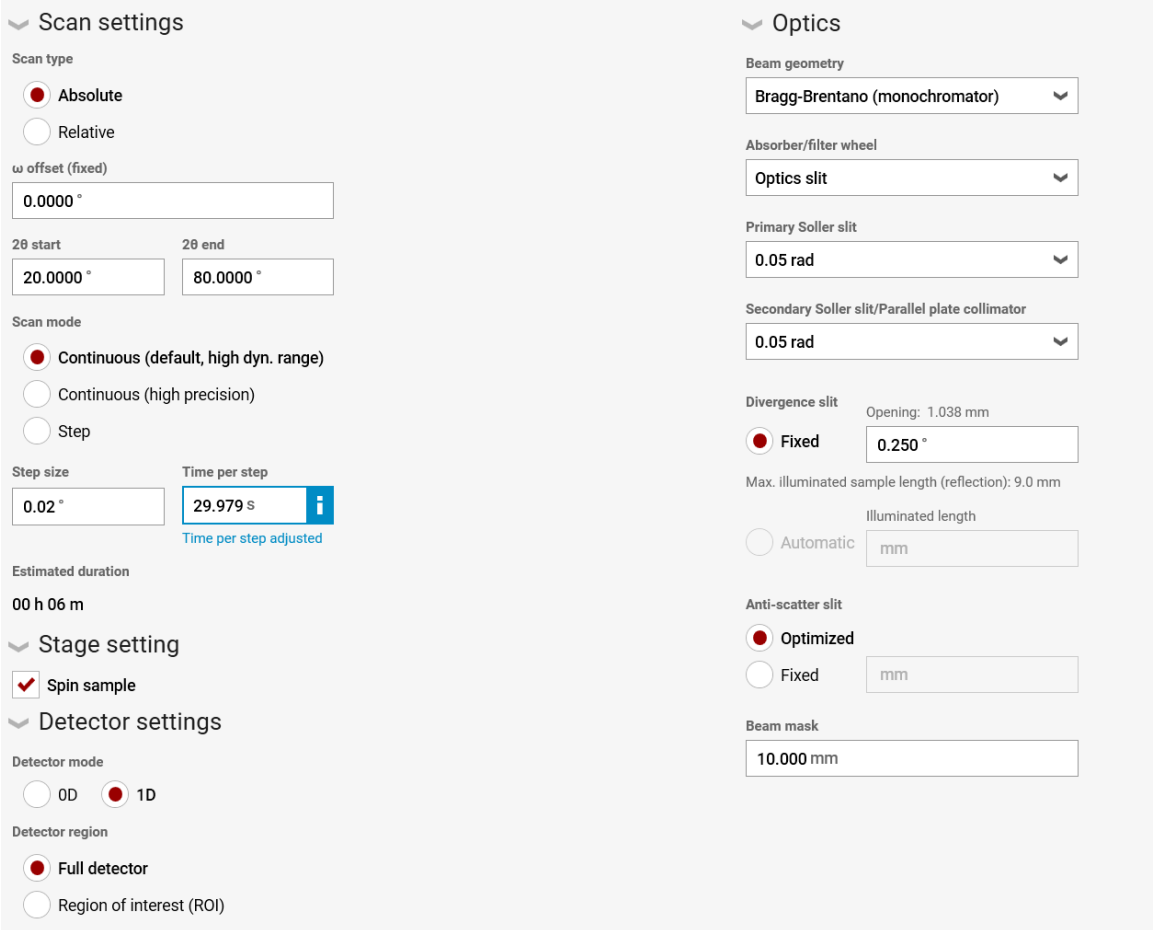


- Connect the turbo pump power cord and pressure gauge cord
- Turn on the pressure gauge and roughing pump
- Turn on the turbo pump
- The pressure should get down to the low 10-4 mbar range



13. Close the XRD chamber doors
14. In the XRDdrive software, select the “Stage Spinner” option 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:

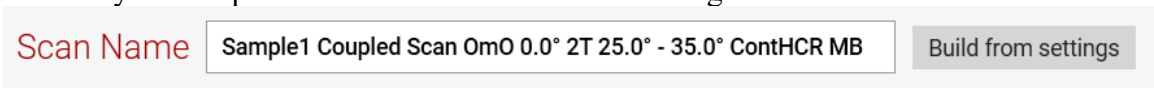


Scan Type: Absolute
w offset: 0.0000
Scan Mode: Continuous
Step size: 0.01 or 0.02 degrees

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

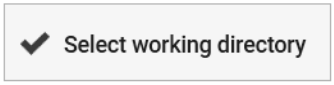
21. Save Measurement Plan by clicking on the menu icon and choosing Save Measurement and then selecting your folder in the XRDExperiments folder

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.

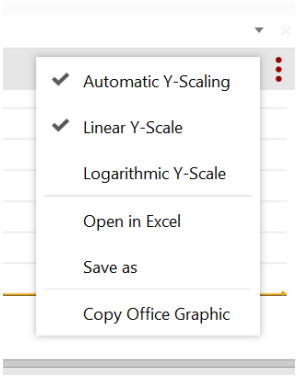


27. Selecting a scan will show the meta data for that scan in a side window
28. Select multiple scan by holding down Ctrl
29. Click the Select box in the bottom right corner to view the scans

- 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 the turbo pump assembly was used:
    - Turn off the turbo pump
    - 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 HTK 1200N stage and remove the adapter. Replace caps on connections.
37. Wait until Sample Stage Temperature is less than 50C
38. Disconnect the cooling water lines
39. Disconnect the HTK 1200N Connector Box
    - Disconnect one end of thermocouple from the Connector Box and the other end from the HTK 1200N stage. ***Handle carefully as the thermocouple wire and connections are fragile***
    - Disconnect flange control from the Connector Box
    - Disconnect spinner switch from the Connector Box
    - Disconnect the box from the XRD chamber
    - Disconnect the heater power supply cable
40. Unload the sample holder from the HTK 1200N stage
    - Remove sample from the stage

- Reinsert sample holder into the HTK 1200N stage

41. Unmount the HTK 1200N stage
42. Remount the Sample Spinner Stage
43. Close the XRD chamber doors
44. In the XRDdrive software, a window should appear indicating that the Sample Spinner stage is mounted
    - Click on “Load Existing Stage Alignment”
45. Turn off X-Rays (if not already off)
46. Close Software
47. Turn off power on front panel
48. Stop your XRD2 reservation in CoreResearch