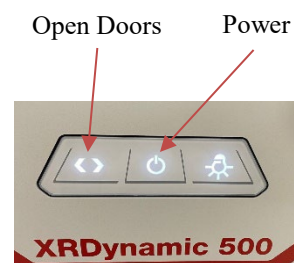


XRD2 Operating Procedure – Sample Spinner Stage

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

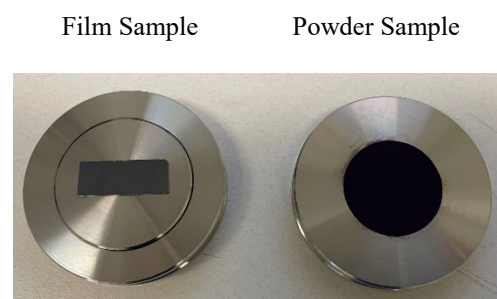
Start-Up

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
 1. Wait until X-Rays have ramped up to 40kV and 50mA before proceeding



Experiment Setup

7. Press the Door Open on the front instrument panel and then slide doors open
8. Load the Sample Spinner Stage (if not already in place)
9. Load your sample into the spinner holder
10. Close the XRD chamber doors
11. Perform Sample alignment
 1. Click on the menu icon and select Alignment from the menu options
 2. Click on the Stage Alignment button and follow instructions
 3. When completed, click close
12. Click on the menu icon and select either
 1. New Measurement Plan (to create a new measurement plan from scratch)
 2. Open Measurement Plan (to open a saved measurement plan and edit)
13. Enter experiment name (the name of your measurement plan)
14. Select the Scan Data Target Folder by clicking on the icon function
 1. Select or create your own folder in the XRDEperiments folder
15. Enter a sample name
16. Build your Measurement Plan
 1. 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)
 - Standard Settings:
 - Scan Type: Absolute
 - w offset: 0.0000
 - Scan Mode: Continuous



- Spin sample: checked for powders, unchecked for films
 - Detector Mode: 1D
 - Detector Region: Full Detector
 - Beam Geometry: Bragg-Brentano (monochromator)
 - Absorber/Filter Wheel: Optics Slit
 - 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
- 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

Scan Name

Sample1 Coupled Scan Om0 0.0° 2 θ 25.0° - 35.0° ContHCR MB

Build from settings

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

2. Select “Add Task” to insert a Wait step or Sample Alignment Step
17. Save Measurement Plan by clicking on the menu icon and choosing Save Measurement and then selecting your folder in the XRDEperiments folder

Experiment

18. Click Start Measurement to run your Measurement Plan
19. The data is automatically saved in two files:
 1. .scn : text file of the raw data (can be opened in Notepad)
 2. .hdf5: file containing the meta data (can be opened in HDFView)
20. When completed, a “Save succeeded” window will appear. Click “Yes” to open the experiment in file explorer and view the scan result

Data Saving, Viewing, and Analysis

Data Viewing (See XRDView guide)

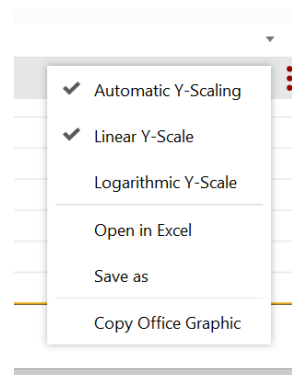
21. Open XRDview
22. Select “Start viewing data”
23. Click “Select working directory” and go to the directory that contains the scans you’d like to view.
24. Selecting a scan will show the meta data for that scan in a side window
25. Select multiple scan by holding down Ctrl
26. Click the Select box in the bottom right corner to view the scans
 1. If multiple scans have been selected, an overlay plot will be generated
 2. Open the side window pane to adjust plotting plot settings
 3. Plots can be saved in picture form by selecting “Export Chart”


 A rectangular button with a light gray background, containing a checkmark icon and the text "Select working directory".


 A rectangular button with a red background, containing a white checkmark icon and the text "Select".


Data Analysis (See *XRData analysis guide*)

27. Open XRData analysis
28. Create New Project
29. Add Measurement Data (select the hdf5 file you would like to analyze)
30. To save the raw data to Excel:
 1. Click the 3 dots in the upper right corner and select “Open in Excel”
 2. Save the Excel file to your folder in the XRDataExperiments folder
31. Perform Data Analysis if desired
 1. Search peaks
 2. Fit peaks
 3. Match (to files in ICDD database)
 4. Quantitative Fit

**Shut down**

32. Press Door Open on the front instrument panel and then slide the doors open
33. Unload your sample
34. Close the XRD chamber doors
35. Turn off X-Rays (if not already off)
36. Close Software
37. Turn off power on front panel
38. Stop your XRD2 reservation in CoreResearch