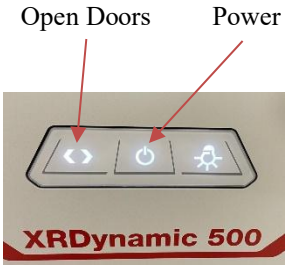
XRD2 Operating Procedure – Sample Spinner Stage

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

Start-Up

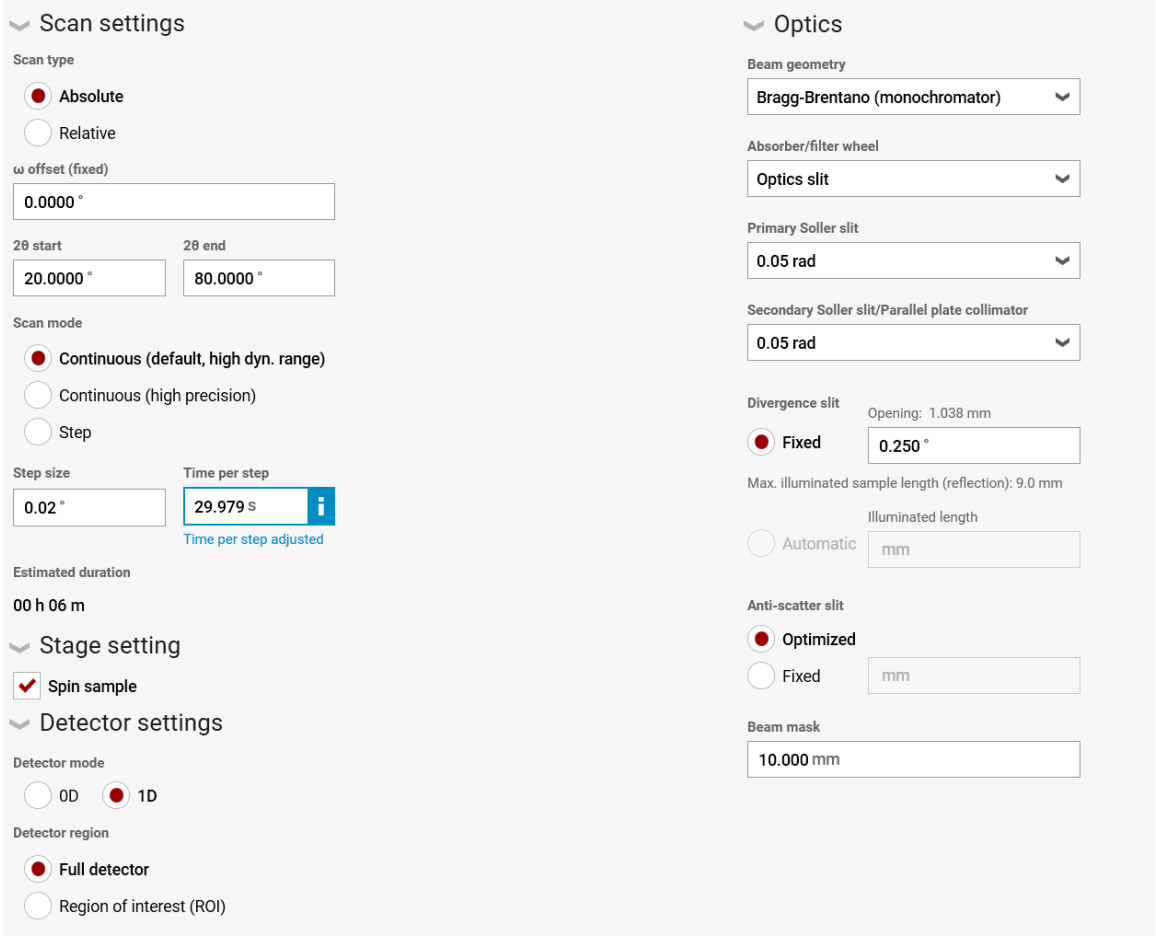


2. Turn on power on front instrument panel
3. Wait until the power button stops flashing and all buttons on the front panel are illuminated (this takes about 1 minute)
4. Open XRDdrive software
5. Click the Connect button
6. Turn on X-Ray source
    1. Wait until X-Rays have ramped up to 40kV and 49mA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

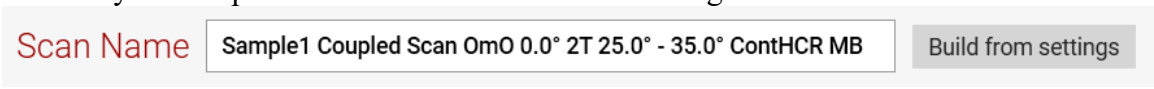
12. Click on the menu icon and select either
    1. New Measurement Plan (to create a new measurement plan from scratch)
    2. Open Measurement Plan (to open a saved measurement plan and edit)
13. Enter experiment name (the name of your measurement plan)
14. Select the Scan Data Target Folder by clicking on the icon function
    1. Select or create your own folder in the XRDExperiments 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)
        - Typical Settings shown on 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

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 XRDExperiments folder

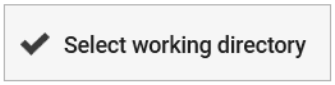
Experiment

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)

Data Saving, Viewing, and Analysis

Data Viewing (*See XRDView guide)*

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

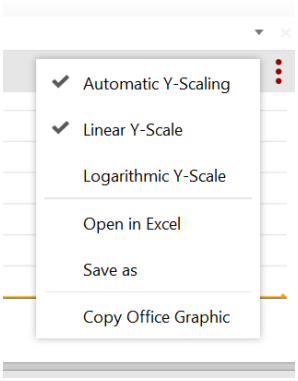


23. Selecting a scan will show the meta data for that scan in a side window
24. Select multiple scan by holding down Ctrl
25. Click the Select box in the bottom right corner to view the scans
    1. If multiple scans have been selected, an overlay plot will be generated
    2. Open the side window pane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

Data Analysis (*See XRDanalysis guide)*

26. Open XRDanalysis
27. Create New Project
28. Add Measurement Data (select the hdf5 file you would like to analyze)

29. 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 XRDExperiments folder
30. Perform Data Analysis if desired
    1. Search peaks
    2. Fit peaks
    3. Match (to files in ICCD database)
    4. Quantitative Fit



Shut down

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