Bruker D8 Scintillator Use Manual
θ-2θ Standard Operating Procedure (SOP)

Basic Parameters

Geo	Start	Stop	Step	Time	Detector	Det Slits	Det Optics	Det Filters	Tube Optics	Tube Slits
θ-θ	10°	90°	0.05°	1s/step 3deg/min	Scint	8mm	0.23 Soller	N/A	Göbel Mirror	0.6mm, Snout
ω @ 39.95 2θ	13°	27°	0.02°	1deg/min	Scint	8mm	0.23 Soller	N/A	Göbel Mirror	0.6mm, Snout

Swapping Detector Optics

1. Carefully loosen LynxEye Bolts (See Below)
2. Slide LynxEye of he XRD Arm
3. Side the Scintillator onto the XRD Arm
4. Carefully tighten Scintillator Bolts
5. In *XRD Commander Details* tab select *Detector 1* and click *Set Detector*

Install and Align Soller Slits

1. Fks
2. Park the tube and detector at 17.569 (35.138 divided by 2).
3. Open the Ratemeter and adjust the Soller Slits screw to maximize intensity
    a. About 12,000 CPS for New tube

001 - 21.824
002 - 44.590

Remove PSD detector arm by loosening the two chrome fasteners on the bottom of the PSD detector arm furthest towards the back of the XRD system and carefully guiding the arm off the end of the slide grooves. Place the detector on the foam pads on the operator's right of the XRD enclosure.
2. Install the detector 1 arm found on the far operator's right of the XRD enclosure by loosening similar silver fasteners and sliding the arm down the slide grooves until the arm sticks on the stop screw. Tighten the silver fasteners until the arm is stable but not so much they cannot be loosened after the measurement is complete.
3. Install the solar slit filter in front of the detector by loosening the black knob and slipping it onto the slide grooves. (Note: this does not need to slide along the slide grooves initially it simply slips onto them) Ensure the filter is installed so the black tightening knob points towards the bottom of the system and the two metal plates face the detector arm. Position the solar filter so that it is only a few millimeters in front of

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the detector but ensure it is not actually making contact.
4. Solar slit alignment
a. In XRD commander uncheck all drives with the exception of tube and detector.
b. Set both tube and detector to 17.569 and click move drives button.
c. Place the 1976 XRD flat plate intensity standard on the XRD stage.
d. Use the laser and video programs to adjust the z-drive/stage height the so the laser is in the center of the target displayed by the video program.
e. Under the diffractometer drop down menu select the "Rate Meter" option.
f. Close the doors of the x-ray, select detector 1 in the rate meter and click start.
g. Observe the maximum intensity then click stop
h. Adjust the small screw on the bottom of the solar slit filter (1/25 of a turn at a time)
i. Repeat steps f, g, and h until the intensity is maximized.
j. Remove the intensity standard.
5. Place the sample that is to be measured on the XRD stage
6. Readjust the z-drive using the laser and video program
7. Click the "Details" tab at the bottom of the XRD commander window
8. Click detector 1
9. Click set detector
10. Back on the adjust tab select seconds per step. Set scan speed to 0.5 and increment to 0.02

11. Run a continuous scan from 84.5 to 87.5 (for Pt 222 peak)
12. Save the scan
13. Open the scan in EVA
14. Select the "Area" tab
15. Click create
16. Select an area from 2θ = 85 to 87
17. Record the "chord mid" value from the area tab
18. Input this value into the 2Theta section of the XRD commander
19. Unselect all other drives and click the move drives button
20. Change the scan type from "Locked/Coupled" to "Rocking Curve"
21. Adjust start to 35 and stop to 50 (for Pt 222)
22. Adjust increment to 0.02 and scan speed to 0.5
23. Start the scan
24. Save the rocking curve scan
25. Open the scan in EVA and use the area tab to collect the chord mid value
26. For additional samples repeat steps 11 to 25
27. When done collecting data it is your responsibility to remove the detector 1 arm and solar slit filter and reinstall the PSD detector arm.
28. Once the PSD arm is reinstalled click the details tab in the XRD Commander program select the PSD detector and click the set detector button.
29. Run a brief locked coupled continuous scan to ensure the PSD arm is properly installed

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and initialized.
30. Remove samples and clean the stage/system if necessary. If the stage was changed reinstall the original metal clip stage.

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