

Reciprocal Space Maps

1. Log into the User Log System on the SMIF web site
2. Follow steps 1 – 26 from the “**Rocking Curves**” procedure to set up the hardware and align the sample.
3. ***Diffracted Beam Optics***
 - Mount Detector 1 into the triple axis location (Back position) of the “rocking curve + triple axis” optics on the diffracted beam side.
4. Select the **Diffracted Beam Optics** tab.
 - Right click on the triple axis heading and select “activate” in the box that appears to move the detector to the triple axis position
 - Double click on any item, and open the Detector tab and make sure that Proportional detector Xe[2] is selected.

Detector position optimization

5. Select **Manual Scan** from the **Measure** pull down menu, and enter the following parameters for a 2theta alignment:
 - **Scan Axis:** 2Theta
 - **Range:** 1.5
 - **Step Size:** 0.005
 - **Time per step:** 0.2Click the *start* button to begin the scan
6. After the measurement is completed find the peak in the scan data and move the goniometer to this selected position by right clicking and using the “Peak” mode

Setting the attenuator

7. Select the incident beam optics tab and double click on any item. Make the following changes in the incident beam optics settings window:
 - **Beam Attenuator:**
 - **Usage:** At pre-set intensity
 - **Activate at level:** 550000
 - **De-activate at level:** 400000Click OK to apply these settings and close the window

Determining the measurement range

Omega-2theta

If the desired omega-2theta measurement range is already known (e.g. from a previous rocking curve), then skip to step 10

8. Back in the **manual scan** window, enter the follow parameters:

- **Scan Axis:** omega/2Theta
- **Range:** 5
- **Step Size:** 0.005
- **Time per step:** 0.2

Click the *start* button to begin the scan

9. Note the desired range (the range over which counts above background occurs)

Omega

If the desired omega range is already known then skip to step 13

10. Back in the *manual scan* window, enter the follow parameters:

- **Scan Axis:** omega
- **Range:** 3
- **Step Size:** 0.002
- **Time per step:** 0.2

Click the *start* button to begin the scan

11. Note the desired range (the range over which counts above background occurs)

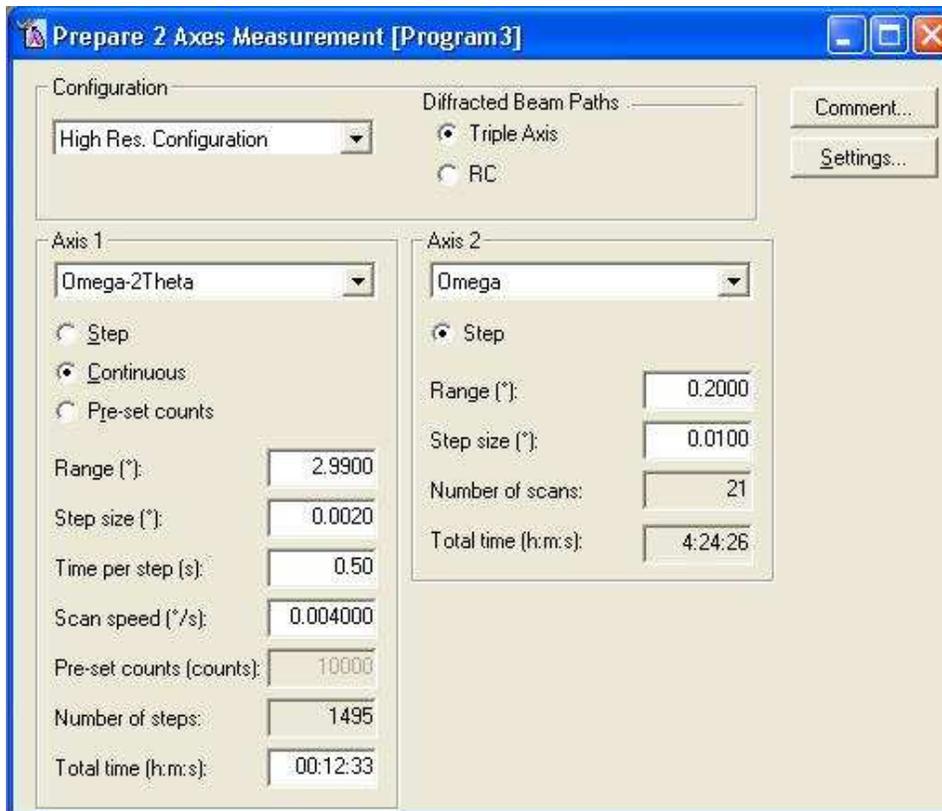
12. Close the display manual scan and the scan dialog windows.

Measurement Program

13. Create a new measurement program or edit an existing program if needed.

- To create a new measurement program:
 - Select **File/New Program**
 - Select Program Type: **2-axis scan**
 - Click OK
- To load and edit an existing program:
 - Select **File/Open Program**
 - Click “Browse” to search for your measurement program
 - Select desired program from the list and click OK

14. The window shown below will appear. Enter the information as explained below



- **Configuration:** High Res. Configuration
- **Diffracted Beam Paths:** Make sure that the triple axis is selected
- **Axis 1:** Omega-2theta
- **Axis 2:** Omega
- **Ranges:** Enter values based on previous results or the ranges noted in steps 9 and 11.
- **Step Sizes:** Values should be small enough to have at least five steps across the narrowest feature.
- **Time per step:** Use 0.5 seconds/step or greater

15. Select **File/Save as** and enter a name for the program and then press the *OK* button.
Close this window.

Measuring

16. Select **Measure/Program**
 - Click “Browse” to search for your measurement program
 - Select the desired measurement program from the list and click Open
17. A start menu will appear
 - Click on the folder icon and select or create a folder where the data will be stored.
(Your data should be stored in your own folder within the XRD Data folder)
 - Enter a filename (sample name)
 - Click the Save button
 - Back in the start menu, enter comments and sample ID if desired
 - **Do not modify any of the Position fields or Reflection fields.**
 - Click the OK button and the measurement will begin
18. When the measurement is completed, you can view and process the data using the X’Pert Data Viewer Program
19. For subsequent samples, return to the *Sample Mounting and Alignment* section from the “**Rocking Curves**” procedure to mount and align the next sample.

Shut Down

20. Unmount your sample from the stage and remove any tape left on the stage
21. Select the **Instrument Settings** tab. Double click on the **Generator** settings. Return the generator setting to the idle values:
 - **Tension:** 45 kV
 - **Current:** 20 mAClick OK to apply these settings and close the window
22. Exit the software
23. Log out of the User Log System on the SMIF web site