

Diffraction Space Maps

1. Follow steps 1 – 37 from the **Rocking Curve** instructions.
2. Select the diffracted beam tab in the control window.
3. Right click on the secondary beam path and select triple axis in the box that appears.
4. Back in the *manual scan* window, enter the follow parameters: 2theta in the *scan axis* field, enter 0.005 in the *step size* field, 1.5 in the *range* field, and 0.2 in the *time per step* field. Press the start button.
5. After the measurement is completed press the right mouse button and select **Move mode**.
6. Press and hold the left mouse button until the cursor is placed over the center of gravity of the peak. This will move the goniometer to the selected position.
7. Back in the *manual scan* window, enter the follow parameters: omega/2theta in the *scan axis* field, enter 0.005 in the *step size* field, 5 in the *range* field, and 0.2 in the *time per step* field. Press the start button.
8. Note the range over which counts above background occur.
9. Back in the *manual scan* window, enter the follow parameters: omega in the *scan axis* field, enter 0.002 in the *step size* field, 3 in the *range* field, and 0.2 in the *time per step* field. Press the start button.
10. Back in the control window select the incident beam optics tab. Double click on an item related to the beam attenuator. Change the usage field to 'switch at preset intensity', set the activate level to 450,000, and the deactivate level to 400,000. Make sure the attenuation factor is correctly set. If it is not change the value using the menu item **Customize/Beam Attenuators**.
11. Note the range over which counts above background occur.
12. Close both the display manual scan and the scan dialog windows.
13. Select **File/New Program/2-axis scan**.
14. The window shown below will appear.

Prepare 2 Axes Measurement [Program1]

Configuration: HR line

Secondary beam path: ☒ Lower ☐ Upper

Comment...
Settings...

Axis 1

Omega-2Theta

☐ Step
☒ Continuous
☐ Pre-set counts

Range (°): 2.9900

Step size (°): 0.0020

Time per step (s): 0.50

Scan speed (°/s): 0.004000

Pre-set counts (counts): 10000

Number of steps: 1495

Total time (h:m:s): 00:12:28

Axis 2

Omega

☒ Step

Range (°): 0.1900

Step size (°): 0.0100

Number of scans: 20

Total time (h:m:s): 4:09:10

Make sure that the triple axis is selected as the secondary beam path.

The scan axes (1 and 2) should be specified as shown above.

The ranges should be picked based on the ranges noted in steps 8 and 10.

The step sizes should be small enough to have at least five steps across the narrowest feature.

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

15. Select **Measure/Program/2-axis scan**.

16. Enter the program name chosen in step 14 if it does not already appear. Enter a data set name and sample identification. **Do not modify any of the angular fields, the HKL field or the Unit cell field.** Press the start button.