

Phase Analysis

(grazing incidence)

Hardware Setup

1. The line source of the x-ray tube must be used. The 1/32 degree slit must be set for sample alignment. A 1/8 degree slit should be used for data collection. An appropriate beam mask must be selected based on the sample size.
2. The 0.27 degree parallel beam optics must be mounted on the diffracted beam side.
3. **Make sure that the collimator slit is present behind the collimator.** Also make sure the detector is mounted behind the parallel plate collimator. The top of the detector mount should be flush with the mount on the parallel plate collimator.

User Setup

4. Open the **Organiser** program.
5. Enter your user name and password. (see page 3-3 in the X'Pert Organiser User's Guide to create a new user if you do not have a user name.)
6. Select the **Users & Projects** menu.
7. Select **Select Projects** to select an existing project or **Edit Projects** to create a new project. (See page 3-10 in the X'Pert Organiser User's Guide for more information on creating new projects.)
8. Select **Modules/X'Pert Data Collector** or the Data Collector button on the tool bar to open the Data Collector Program.
9. Select **Instrument/connect** in the Data Collector. The Go On Line box will appear.
10. Select the PDS + PPC that defines the selected optics setup. Then press the OK button.

Optics Setup

11. Selected the incident beam optics tab. Double click any item in the tree view to activate a new window. Change the optics to reflect the current instrument setup. The mirror must be selected, a 1/8 degree divergence slit should be selected even though a 1/32 is currently in place, and if a beam mask is used the correct size should be selected. The beam attenuator should be set to 'do not switch' and activated.

12. Select the diffracted beam optics tab. Double click any item to activate a new window. Select the appropriate optics to reflect the settings.

13. Select the Instrument settings in the main window. Select the x-ray tab. Set the generator to 45 kV and 40 mA. Then, press apply.

Direct Beam Intensity Determination

14. In the same window press the *Change position* tab and enter 0 in the *2theta* field, 0 in the *offset* field (this sets omega to 0), 30 in the *phi* field, 0 in the *psi* field and 0 for x, y, and z. Press the *OK* button to move to these positions.

15. Select **Measure/Manual Scan** from the main menu.

16. Enter the follow parameters: 2theta in the *scan axis* field, enter 0.01 in the *step size* field, 3 in the *range* field, and 0.1 in the *time per step* field, then press the start button.

17. After the measurement is completed press the right mouse button and select **Move mode**.

18. 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. Note the current intensity. (Write it down.)

Bisecting the Beam with the Sample

19. Mount the sample on the stage. The long direction should be horizontal.

20. Press the *Change position* button. Adjust the z position of the sample stage until the counts read between ten and twenty percent of the value noted in step 18. Then press *OK*.

21. Back in the **Manual Scan** window, enter the following parameters: omega in the *scan axis* field, enter 0.005 in the *step size* field, 2 in the *range* field, and 0.1 in the *time per step* field, then press the start button.

22. After the measurement is completed press the right mouse button and select **Move mode**.

23. 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.

24. If the current intensity is now **half** (within 1 percent) of the intensity noted in step 18 continue with step 25. If it is more or less than half then return to the **Control** window. Increase z if the intensity is higher than half the value or decrease z if the

intensity is lower than half the value. Adjust z so the intensity is half the value of that noted in step 18 and then return to step 21.

25. Select **Sample Offsets** from the menu. Enter zero in the omega field.
26. Back on the incident beam tab of the control window set the attenuator to 'preset intensity'. The activate level should be 450,000 and the deactivate level 400,000. Press the *OK* button. Make sure that the attenuation factor is set to 149. If it is not select Customise/Beam Attenuators and set the factor of the Ni 0.125 mm attenuator to 149.
27. Change the slit size from 1/32 degree slit to 1/8 degree slit. Remove the collimator slit.

Measurement Programs

28. Select **File/New Program/Absolute scan**.
29. Enter the information in the fields as explained below. Choose 2θ as scan axis. Select an omega value (0.5 degree, 1 degree, etc.). Choose continuous mode. Input range, step size, time per step, etc.
30. Select **File/Save as** and enter a name and description for this scan program and then push the OK button. Close this window.

Measuring

31. Select **Measure/Program/Absolute scan** and the program name used in step 30.
32. Enter a data set name and description. Enter the desired value for the incident angle in the omega field. Press the start button.