

CHAPTER 5

USING DATA COLLECTOR

WITH X'PERT PRO MPD

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5.1 INTRODUCTION

In this chapter we are going to perform two measurements:

- one on the "standard" (delivered with the system) silicon sample
- the other on a capillary sample of your own choice

5.1.1 System

The system that we will use to gather the data is an X'Pert PRO crystallography configuration for phase analysis comprising:

Sample Stages:	Multi-purpose sample stage Sample spinner Capillary spinner
Incident Beam Optics:	Curved monochromator X-ray mirror Hybrid monochromator Programmable optics
Diffracted Beam Optics:	Programmable optics Parallel beam collimators Position sensitive detector Double optics arm.

5.2 SYSTEM PREPARATION

5.2.1 Starting the Software

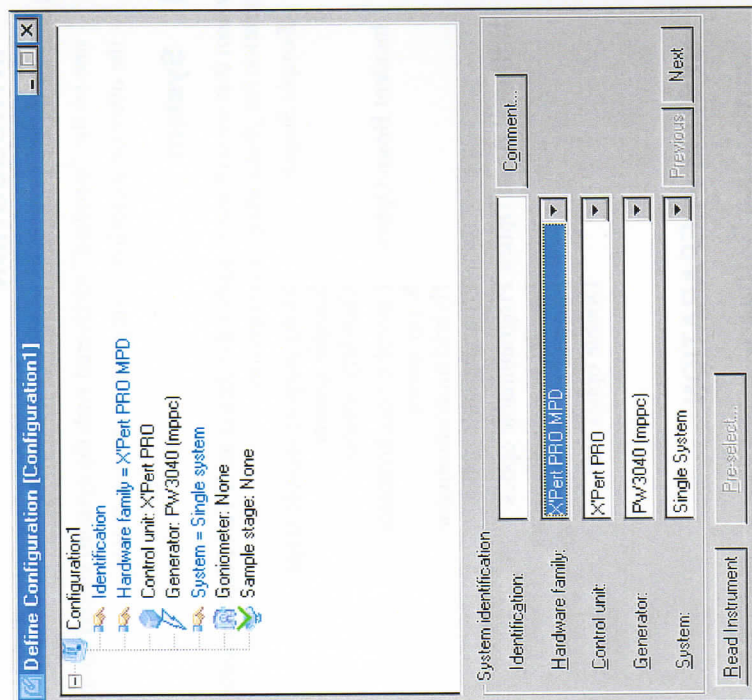
- Double-click on the Data Collector icon, enter your user name and password and press .

5.2.2 Describing your Hardware

Before you can start to collect data you have to tell the software what hardware is used in your diffraction system.

- Select *File - New Configuration*.

In the "System identification" frame select your Hardware family (X'Pert PRO MPD in this example).



Find out what hardware is known to the instrument control software by pressing **Bead Instrument**.

Wait until the system is ready (the "Connecting to instrument" pop-up disappears). If you have a double system, select "Left" or "Right" corresponding to the goniometer that you are going to use from the "System" drop-down list. If you have a single system this choice will not be available to you.



Press the **Next** button.

If you have more than one sample stage available to you, tick the "Get all sample stages in instrument" check box in order to reduce the number of sample stages in the drop-down list of sample stages to only those available.

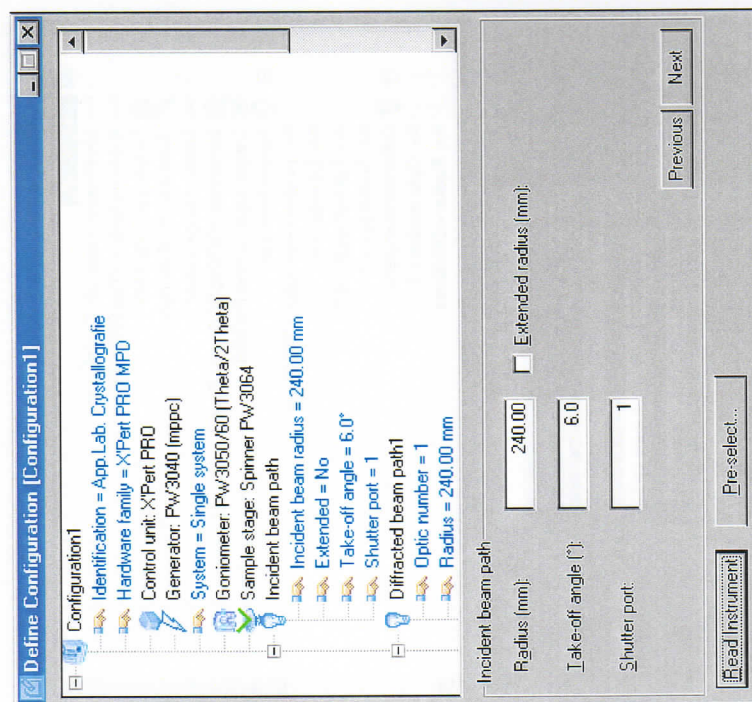
Press the **Next** button to see the Incident Beam Path description.



Confirm that the information displayed here is correct (don't forget to check all diffracted beam paths using the drop-down list).

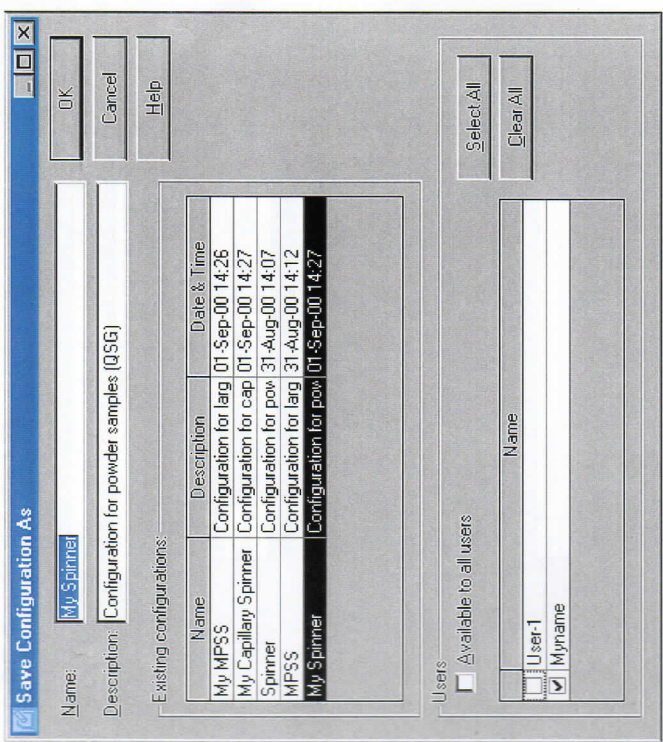
Press the **Next** button to see the "Axes" frame which contains the limits and rest positions which we will not change at this time.

- Press the button **Pre-select...** of pages where you can tell the system what changeable devices are available to you. Always start with the PreFIX module products (the default) if any are available.

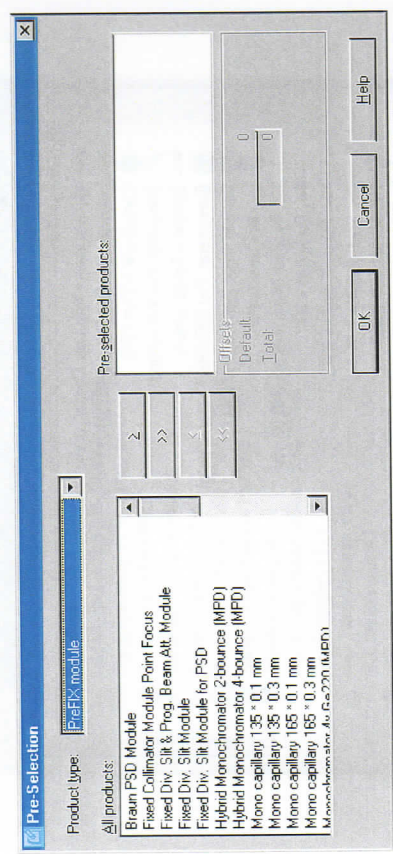


Confirm that the information displayed here is correct.

Press the **Next** button to see the diffracted beam path description(s).



- Press .
- If you have more than one sample stage (for example a spinner and MPSS), you must create a configuration for each sample stage. In this case where we have three sample stages, do the next configuration; go to the "Goniometer/Sample Stage" frame by pressing the button three times.
- In the "Goniometer/Sample stages" frame select the next sample stage from the drop-down list.
- Press in order to inform X'Pert Data Collector about the sample stage offsets for the selected sample stage.
- Save the new configuration by selecting *File - Save As ...* using an appropriate name, in this example: "My MPSS". This saves the same configuration again with the exception of the new sample stage.



Leaf through each entry in the "Product type" drop-down list and select those products that are available to you. To select an item, you highlight that item in the "All products" list and then press .

When you have selected all of the items that you require press .

- Save the configuration by selecting *File - Save As ...* In this example we saved the configuration as "My Spinner".

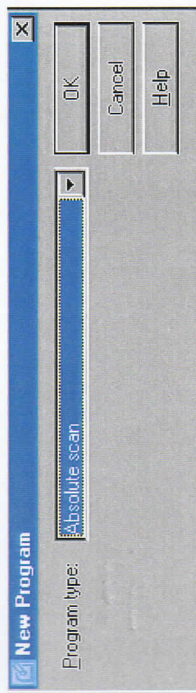
5.3 SILICON SAMPLE

5.3.1 Preparation

- Mount the optical components for your system. In this example we:
 - a. Moved the X-ray tube to the position for use with the incident beam monochromator.
 - b. Mounted the programmable divergence slit PREFIX module.
 - c. Mounted the programmable anti-scatter slit/programmable receiving slit PREFIX module on the diffracted beam side.
- Mount the silicon sample.
- Switch the system on by pressing the "Power On" button on the enclosure's console. When the kV display shows 15 and the mA display 5, the system is ready for use. If the power does not run to 15 kV and 5 mA please refer to your X'Pert PRO System User Guide.

5.3.2 Creating the Measurement Program

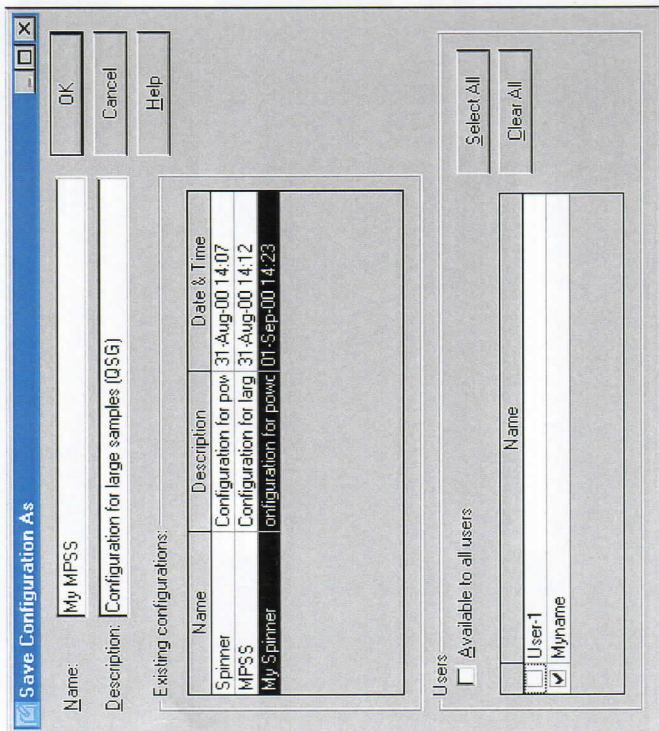
- Select *File - New Program*.



Select "Absolute scan" from the "Program type" drop-down list and press .

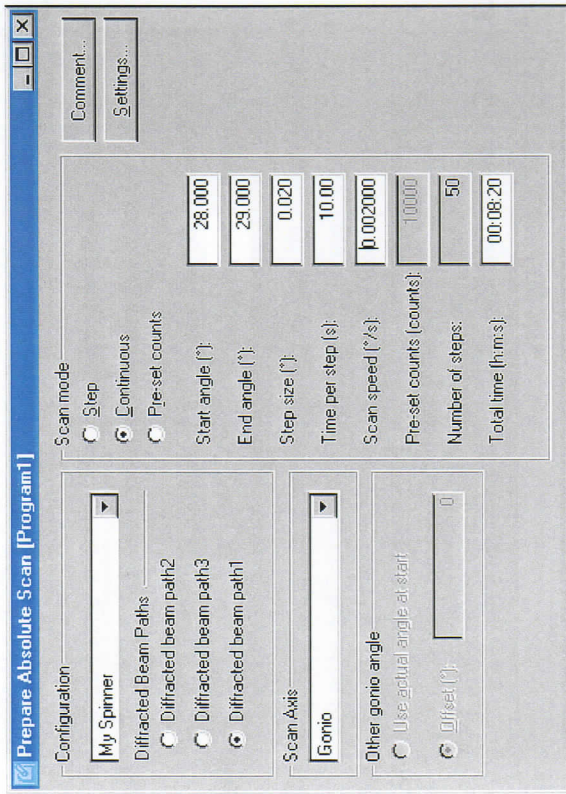
Make the following entries:

Configuration	"My Spinner" (from the drop-down list)
Diffracted beam path	Choose the diffracted beam path containing PRS/PASS PREFIX module (in this example: Diffracted beam path 1).
Scan Axis	Gonio
Start angle	28
End angle	29
Step size	0.02
Time per step	10.00



Press .

- Repeat these actions for the remaining sample stage (capillary spinner) and save the configuration (in this example as: "My Capillary Spinner"). Exit this phase by pressing .



Press **Settings...**

Click on the Sample stage and change it from “Not moving” to “Spinning” (radio button) with a revolution time of 0.5.

In the Incident beam path:

Click on “PreFIX modules” and select the “Programmable Divergence Slit Module” from the drop-down list.

Click on the “Divergence slit” and change the usage to “Automatic” with an irradiated length of 10 and an offset of 0.

Click on “Mask” and select “Incident beam mask fixed 20 mm” from the drop-down list.

Click on “Monochromator” and select “Incident beam al reflectivity” from the drop-down list.

In the Diffracted beam path:

Click on “PreFIX modules” and change it to “PRS/PASS module”.

Click on the “Receiving slit” and make the height 0.1.

Click on the “Anti-scatter slit” and select: usage “Automatic” with an observed length of 10 and an offset of 0.

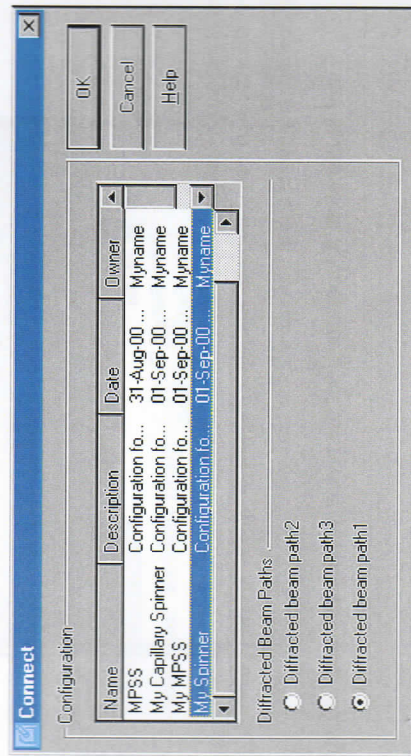
Press **OK**.

- Save the program as “My Monochromator Program” and close the “Prepare Absolute Scan” window by pressing **X**.

5.3.3 Performing the Measurement

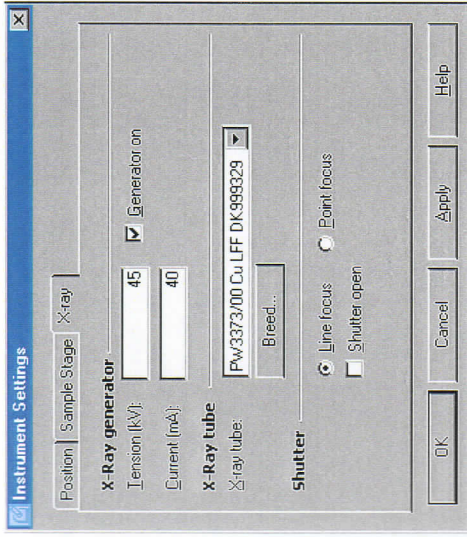
- Select *Instrument - Connect*.

Choose the correct configuration and the required beam path.



Press **OK**.

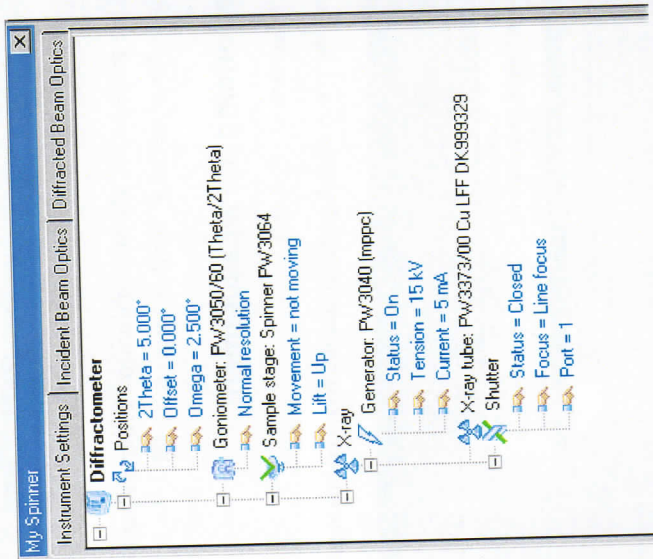
If you get any system instructions follow them.



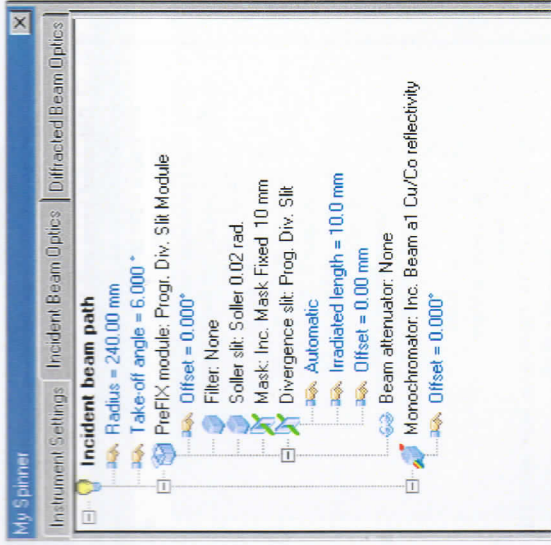
Press **OK**.

- Select the "Incident Beam Optics" tab.

Double-click on the "Incident beam path" and then select the items in the beam path by selecting the relevant tabs and the types from the drop-down lists, starting with the PreFIX module and pressing **Apply** before doing the rest of the changes.



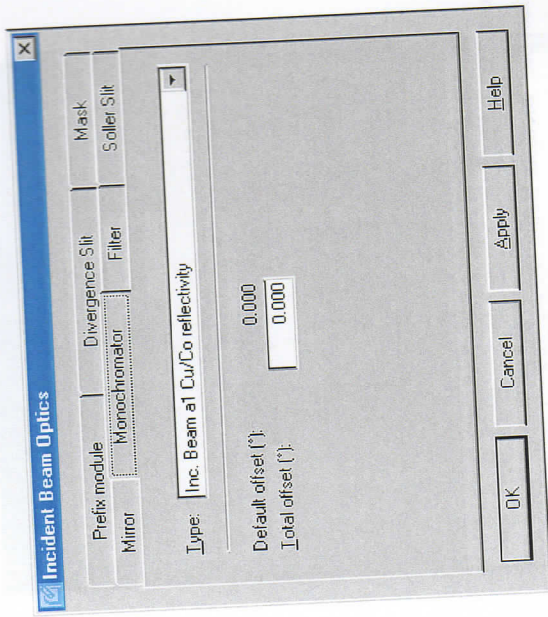
Double-click on "Generator", set the power to "45 kV" and "40 mA".



- Select the "Diffracted Beam Optics" tab.

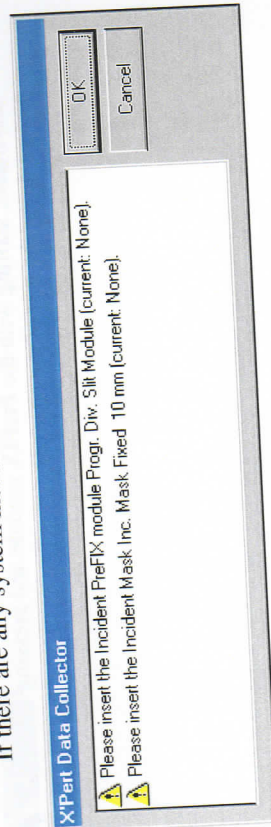
Double-click on the "Active" diffracted beam path and then select the items in the beam path by selecting the relevant tabs and the types from the drop-down lists, starting with the PrefIX module and pressing

Apply before doing the rest of the changes.

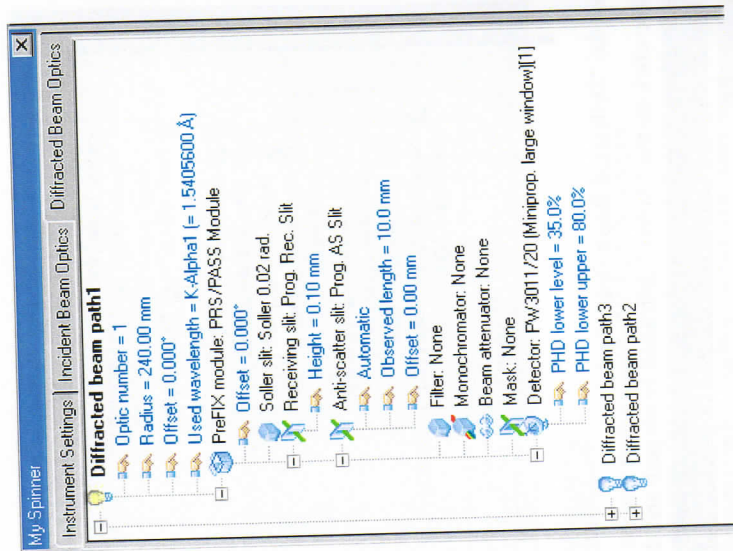


Press .

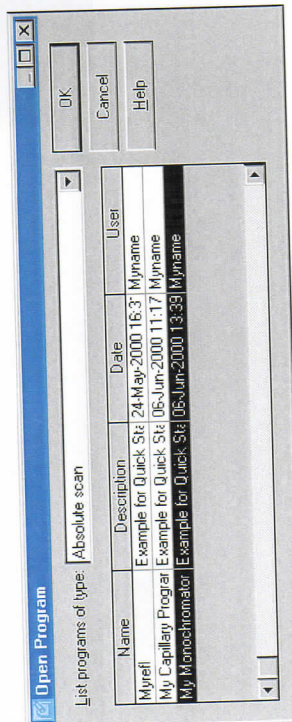
If there are any system instructions, follow them.



Press .



- Select *Measure - Program* and choose "My Monochromator".



Press

Enter a data set name and press again.

The scan results are displayed as the measurement progresses.

