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X'PERT DATA COLLECTOR

Quick Start Guide

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This is the sixth edition of this publication, it is intended for use with version 2.2 of the X'Pert Data Collector software.

ACKNOWLEDGMENTS

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Chapter 1

Introduction

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X'Pert Data Collector - Quick Start Guide

1.1 INTRODUCTION

This Quick Start Guide is intended to help you to use the described X'Pert Data Collector software quickly and efficiently. The examples show you how to start and use the software to perform fairly simple tasks.

In order to follow these examples you must start with Chapter 2, then proceed to either Chapter 3, 4 and/or 5 (depending on your system).

This document is not designed to show in detail or explain all the various possibilities of the software. For each of the examples shown in this document only one route to perform the task is shown, there may be other methods that you can use but you will have to experiment with the system and learn the other possibilities yourself.

NOTE: There may be differences between the example screens given in this Quick Start Guide and what you see on your screen. In all cases, where there is a difference, follow what you see on your screen.

1.2 Contents of the Quick Start Guide

The worked examples in this Quick Start Guide are:

Chapter 2: Starting to Work with X'Pert Data Collector

This chapter describes how to use X'Pert Data Collector for the first time, to create a new user and enable that user to work on the system with all the privileges of a system owner and user manager, and finally how to organize your results.

Chapter 3: Using X'Pert Data Collector with MPD Type Systems

This chapter describes how to use X'Pert Data Collector with Multi Purpose Diffraction type systems to collect data from the silicon sample supplied with the system.

Chapter 4: Using X'Pert Data Collector with MRD Type Systems

This chapter describes how to use X'Pert Data Collector with a Materials Research Diffraction (MRD) type system to measure the silicon (111) single crystal, supplied with the system; and to measure the reflectivity curve of the thin film sample delivered with the system at very shallow (small 2θ) angles.

Chapter 5: Using X'Pert Data Collector with X'Pert PRO MPD

This chapter describes how to use X'Pert Data Collector with an X'Pert PRO MPD crystallography configuration for phase analysis. It describes how to perform two measurements:

- one on the "standard" silicon sample
- the other on a capillary sample of your own choice.

1.3 Terms and Conventions Used

In this section, we describe the terms and conventions used in this Quick Start Guide and how they relate to the user interface.

1.3.1 Terms Used to Denote an Action

In this guide there are several terms that indicate an action.

Check (✔)	Also referred to as a tick mark.
Click	Press the mouse button and quickly release it.
Double-click	Press the mouse button twice (quickly) on an icon, item, file or program.
Drag	Press and hold down the mouse button and move the pointer to define an area or move an object
Enter	Type in information. This can be either text or numerical data.
Press	A key on the keyboard, or a push-button in a window.
Right-click	Press the right mouse button and quickly release it.
Select	Move the mouse cursor to the option you want and click the left mouse button.
Tick (✔)	Also referred to as a check mark.
Toggle	Switch between parameters or states (for example: On-Off-On).
	In the examples in this Guide we terminate most actions by saying “press  ”, you can if you prefer press ***Enter*** instead of  .
	The instruction to click (or press)  is used in this Guide as an instruction to close the window that you are currently working in, **not** the program.

1.3.2 Instructions and Descriptive Text

An instruction is preceded by a bullet "●". Any descriptive text relating to an instruction is given directly after the instruction.

Generally, screen captures are preceded by an instruction and intend to reproduce what you will see on your screen. However, if there are any differences, follow what you see on your screen.

1.3.3 Push-Buttons and Fields

All push-buttons on a dialogue box are shown as the actual push-button (for example: 

), or in bold text (for example: **Apply** or **Cancel**).

All fields are shown between "quotation marks".

1.3.4 Menu Items and Keys

All menu items are printed in italics, for example: *File* - *Open* etc.

All keys are shown bold in an italic font. For example: ***Enter, Ctrl, Alt, Del*** etc.

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Chapter 2

Starting to Work with X'Pert Data Collector

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Chapter 2. Starting to Work with X'Pert Data Collector

2.1 Introduction

This chapter describes how to use X'Pert Data Collector to create a new user and enable that user to work on the system with all the privileges of a system owner and user manager.

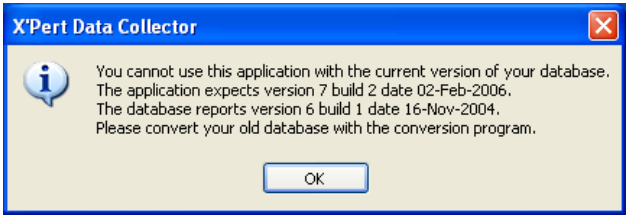
2.2 Starting X'Pert Data Collector

- Double-click on the X'Pert Data Collector icon 

.

  If this is the first time X'Pert Data Collector is used a warning about the database version may appear, if it does proceed with "First Time Use" (2.2.1). If this warning does not appear, go to "Normal Use" (2.2.2).

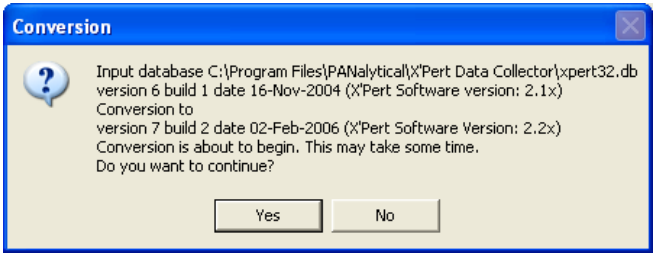
2.2.1 First Time Use



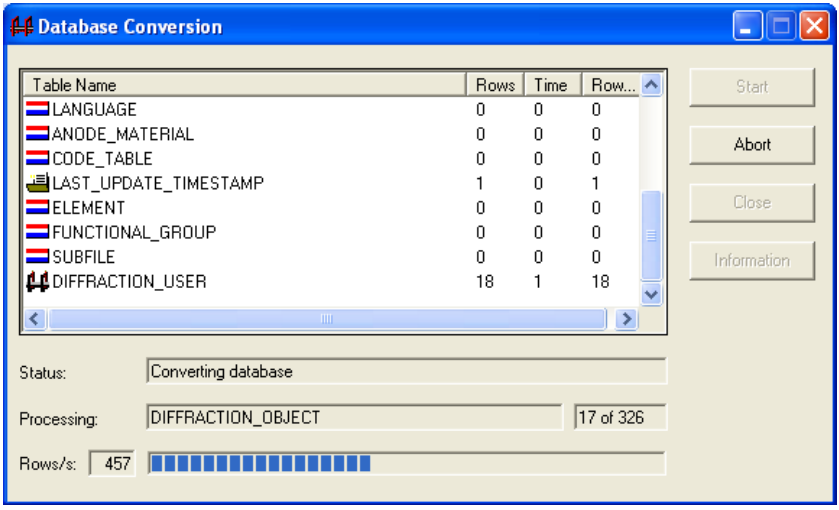
- Press OK.
- Select **Start** – *All Programs – PANalytical X'Pert Data Collector – Utilities – X'Pert Data Collector Database conversion.*
- Select the database to convert (xpert32.db in this example).
- Press Open.
- Press the Yes button to make a backup:

  Either accept the proposed name or rename the database and press Save. If a message about the disk space appears and you have enough disk space press OK.

- When the message about the version appears:

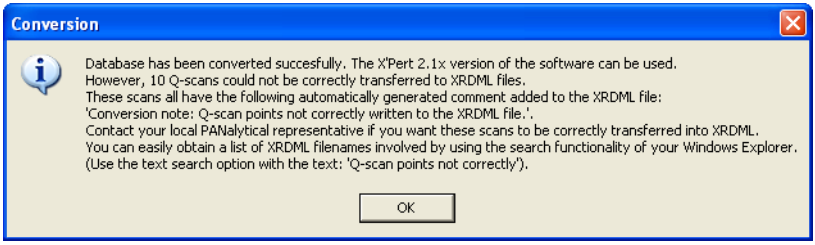


press Yes and the following screen appears:



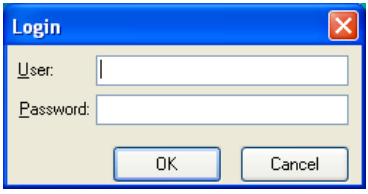
- Press Close as soon as the button is enabled.

You may get a message about incorrect conversion results:



- Press OK.

2.2.2 Normal Use

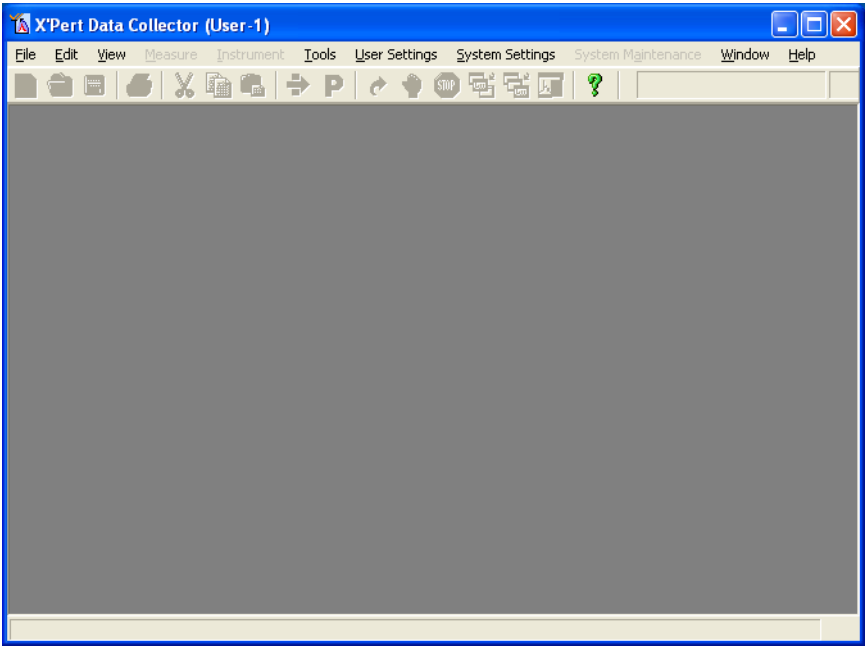


- Log on with the following information:

  User: User-1

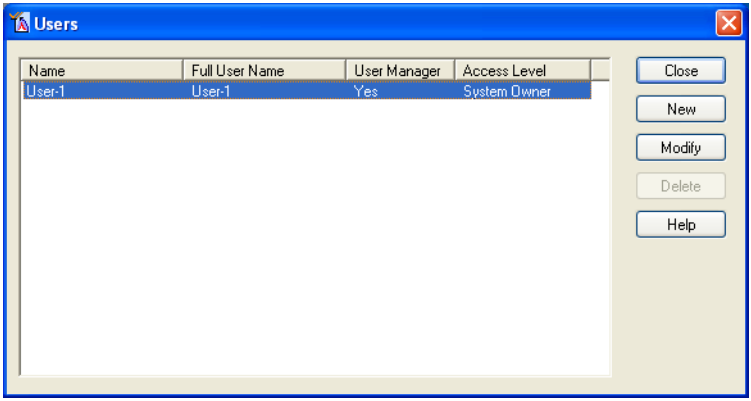
  Password: galaxy

  (the user name and password delivered with the software) and press OK.



In this example we will create a new user.

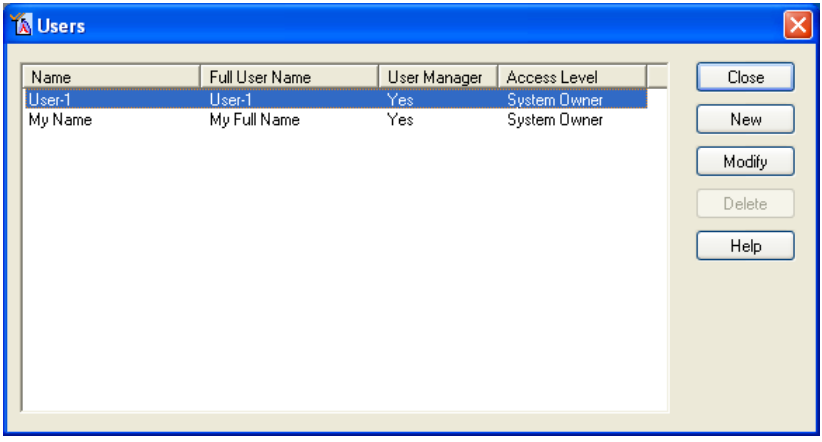
- Select *System Settings – User Management.*



- Press New.

- Enter: Name: = My Name
  Full user name: = My Full Name
  Access level: = Choose "System Owner"
  Check the "User manager" box.
  Password: = password (do not use spaces in your password)
  Re-type Password: = password.

- Press OK.



- Check that "My Name" is included in the "Name" list and press Close.

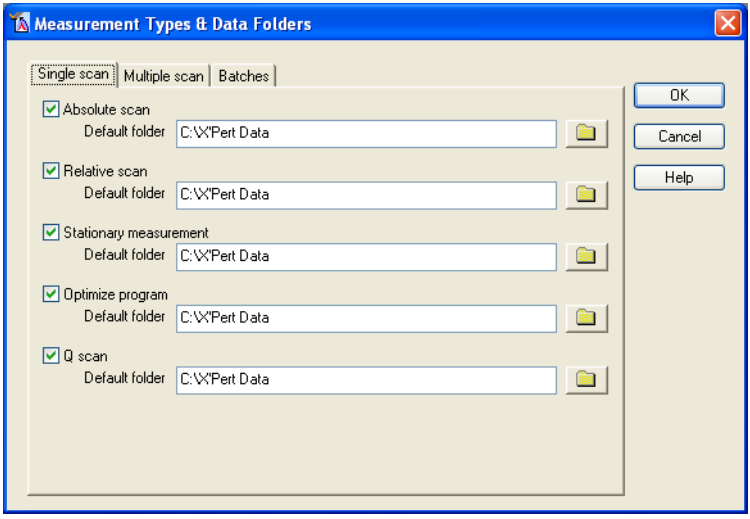
The new user is now created. To start actions as this user you must select *File - Exit* to exit from X'Pert Data Collector and log in again by double-clicking on the X'Pert Data Collector icon:

using the user name "My Name" and the password "password".

2.2.3 Organizing your Result Data

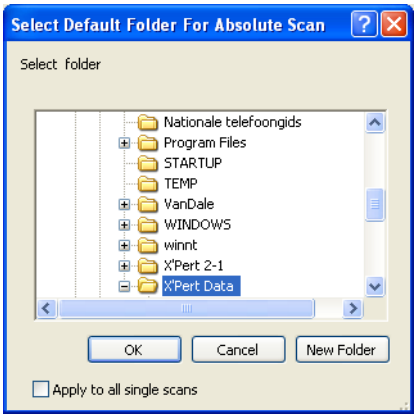
Before you start to use it, you need to tell the system where to put the results of your measurements.

- Select *User Settings – Measurement Types and Data Folders*

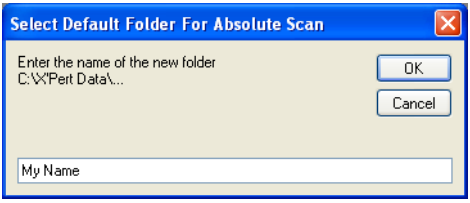


Here is where we define the default folder where all of the results will be placed. If there are types of measurement that you are never going to use you could un-check them here; in this example we will leave them all on.

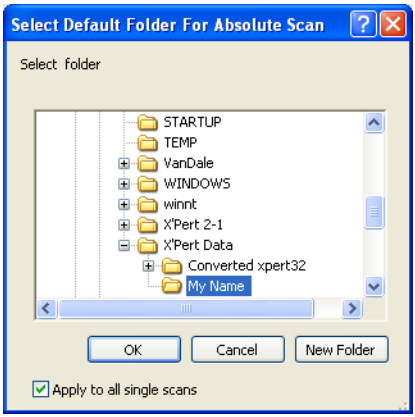
- Press the "Absolute scan Default folder" browse button ( ):



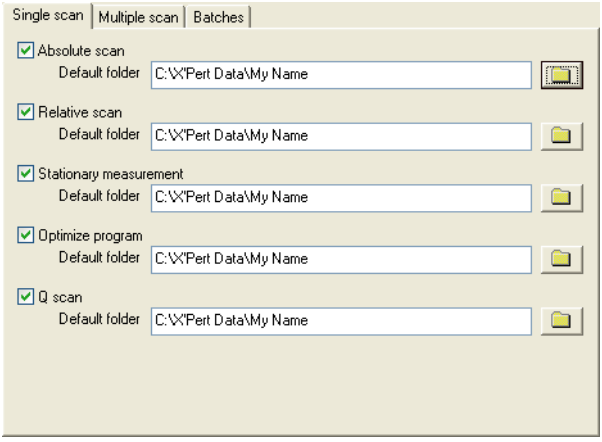
- Press New Folder and enter the name of the folder, in this example: "My Name".



- Press OK.
- Check (tick) "Apply to all single scans".



- Press OK.

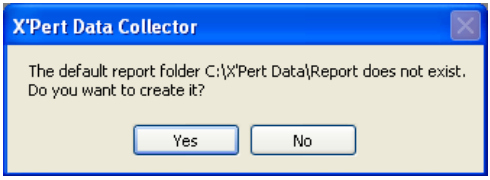


- Repeat these actions for the “Multiple scan” and “Batches” tabs.
- Press OK.

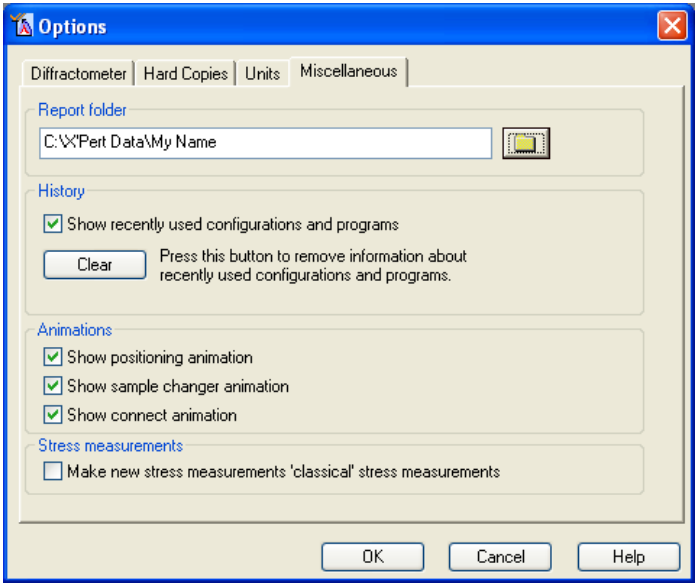
2.2.4 Report Folder

If you create a report of a program or a configuration you can specify the default destination folder.

- Select *User Settings – Options...* and then click on the "Miscellaneous" tab.



- Press No as we will set the system up with respect to the report folder in the following actions.
- Use the button to select the report folder, in this example: "C:\X'Pert Data\My Name".



- Press OK.

The preparation stage is now complete and you can start defining configurations and measurement programs, and performing measurements. To end this part, exit from X'Pert Data Collector, this time by pressing  .

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Chapter 3

Using X'Pert Data Collector with MPD Type Systems

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Chapter 3. Using X'Pert Data Collector with MPD Type Systems

3.1 Introduction

This chapter describes how to use X'Pert Data Collector with Multi Purpose Diffraction type systems (like X'Pert-MPD and X'Pert PRO MPD with normal resolution optics) to collect data from the silicon sample supplied with the system. We presume that you have defined a user "My Name" as described in Chapter 2 of this guide.

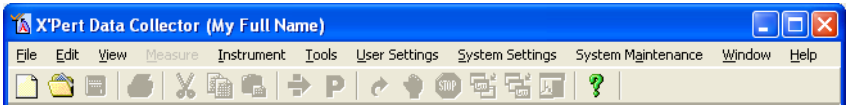
3.2 Preparation

- Switch the system on by pressing the "Power On" button on the diffractometer. When the display shows its start-up values (for example: "30 kV" and "10 mA" for X'Pert-MPD), the system is ready for use. If the power run up does not happen, refer to the relevant hardware User's Guide.

3.3 Starting the Software

- Double-click on the X'Pert Data Collector icon 

.
- Enter the user name and password: "My Name" and "password", and press OK.

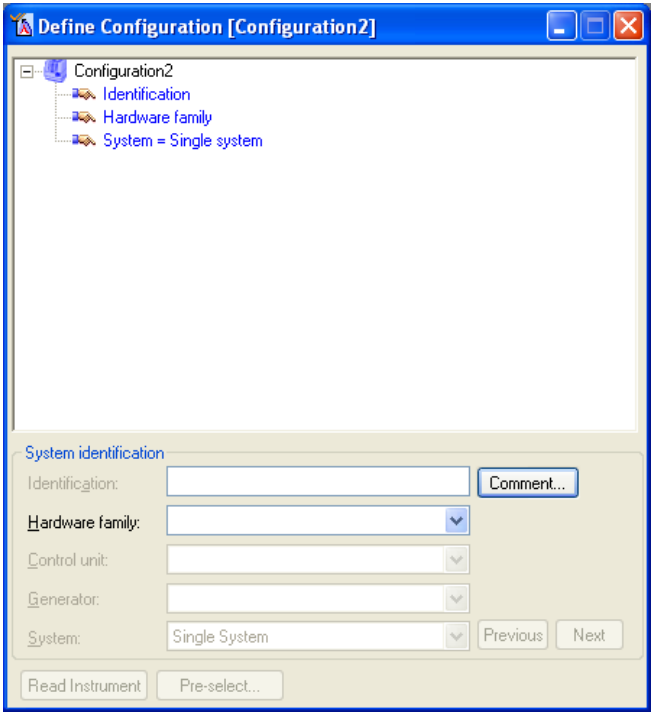


3.4 Describing your Hardware

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

3.4.1 Hardware Family and Configuration

- Select *File - New Configuration...*

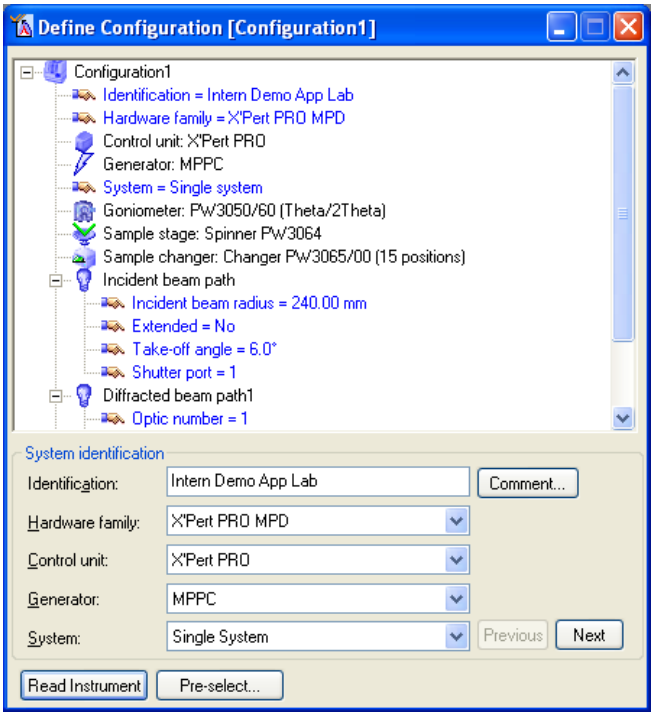


- In the "System identification" frame select your hardware family (X'Pert PRO MPD in this example).
- Find out what hardware is known to the instrument control software by pressing Read Instrument.
  If the system has not been previously initialized an initialization wizard will start. This wizard is designed to handle situations where there

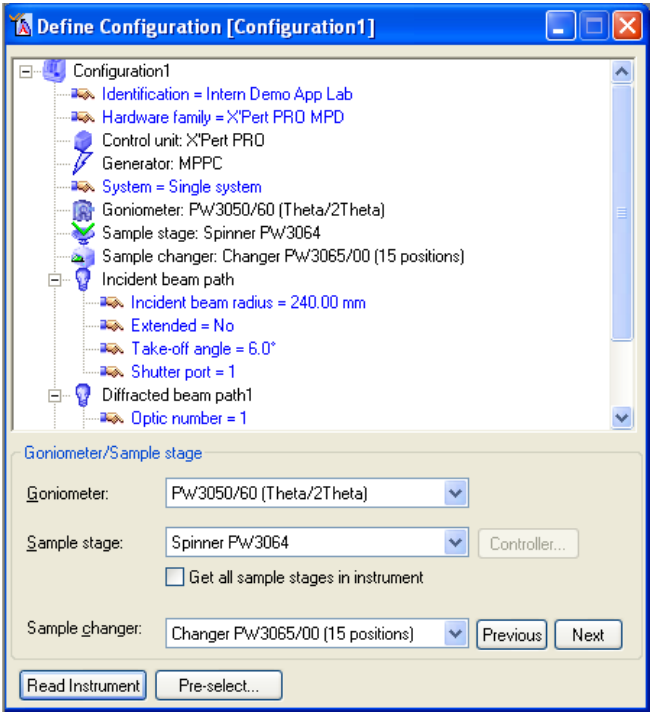
is a possibility of collision when the diffractometer resets (for example: system parts might collide). If this wizard does start, just follow the instructions displayed on the screen.

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.

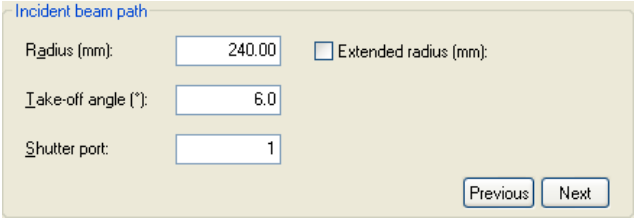
If the system cannot uniquely identify some of the items it will ask you to specify what you have.



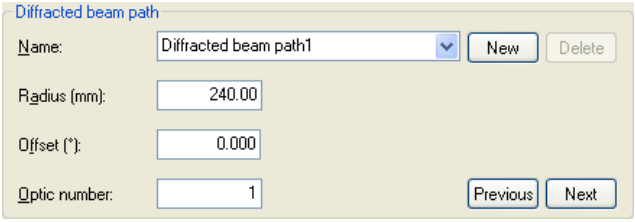
- Press the Next button.



- Press the Next button to see the "Incident beam path" description.

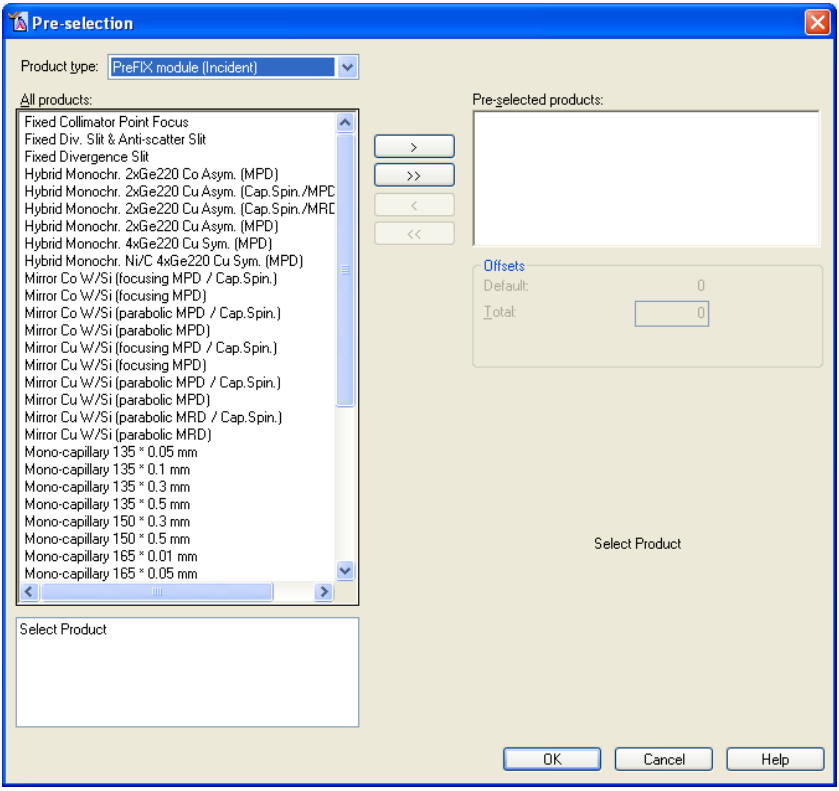


- Confirm that the information displayed here is correct. Press the Next button to see the "Diffracted beam path" description.

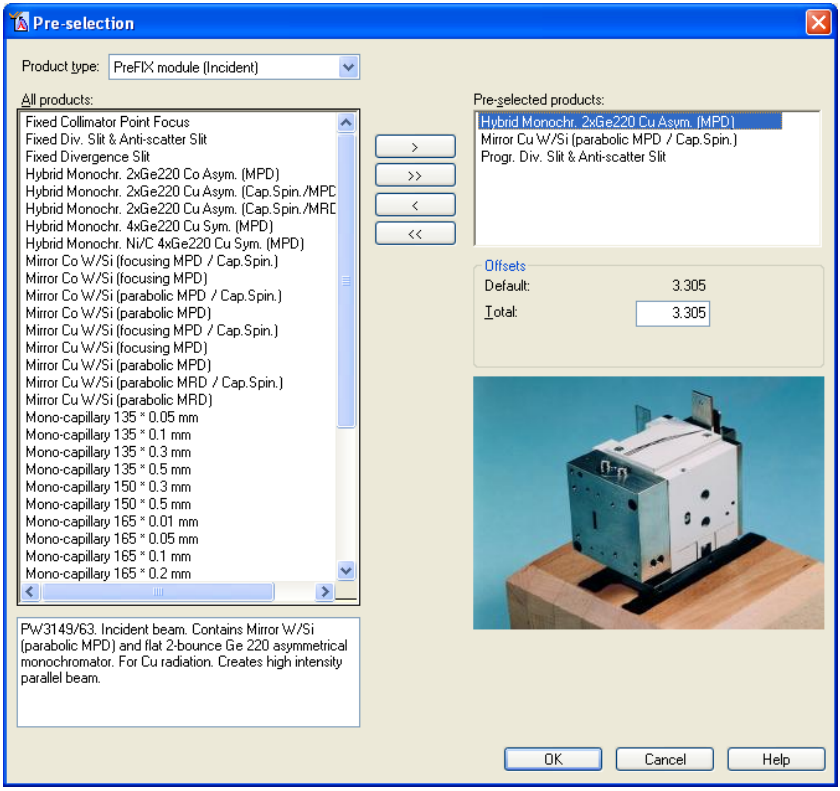


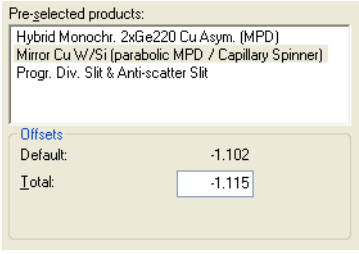
- Confirm that the information displayed here is correct for all diffracted beam paths. Press the Next button to see the "Axes" frame for defining the Limits... and Manual Load Positions... which we will not change at this time.

- Press the button Pre-select... to obtain a series of pages where you can tell the system what changeable devices are available to you. Always start with the PreFIX module products (default is "PreFIX module (Incident)"), then "PreFIX module (Diffracted)" if any are available.



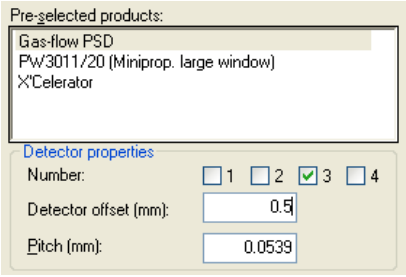
If you have X-ray Mirror or Hybrid monochromator PreFIX modules in your system it is important that you enter their offsets that are provided in the System Acceptance Form delivered with your system.



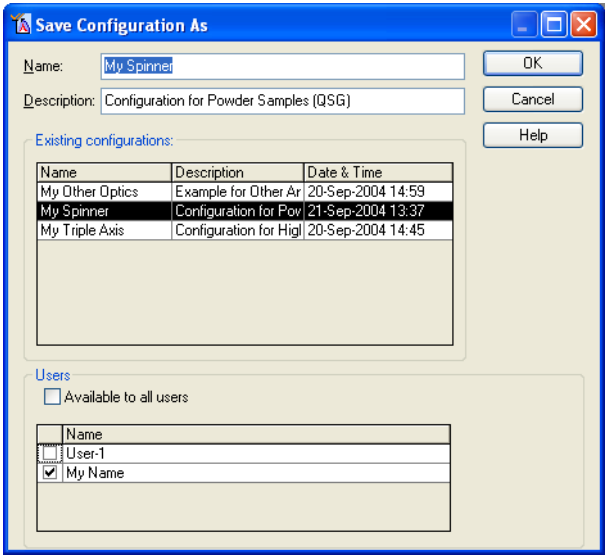


- Leaf through each entry in the "Product type" drop-down list and select those products that are available to you. To select an item, you highlight that item in the "All products" list and then press [>].

If your system includes an X'Celerator detector (and an X'Celerator detector monochromator) or a position sensitive detector it is important to define the detector offsets provided in the System Acceptance Form delivered with your system.



- When you have selected all of the items that you require press OK.
- Save the configuration by selecting *File - Save As...*. In this example we saved the configuration as "My Spinner" and gave it the description "Configuration for Powder Samples (QSG)".



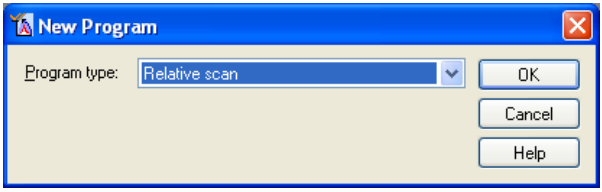
- Press OK and then .

  If you have more than one sample stage you should make a configuration for each sample stage. In order to do that you must mount each of your sample stages in turn and then create a configuration for that stage by repeating the actions in this section (3.4.1). Exchanging sample stages is described in Chapter 5, section 5.4.1.

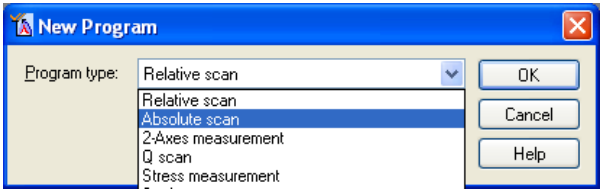
3.5 Defining a Measurement Program

At this stage in the procedure you can either go on-line (connect to the diffractometer), or define a measurement program. In this example we will first define a measurement program and then go on-line.

- Select *File – New Program…*



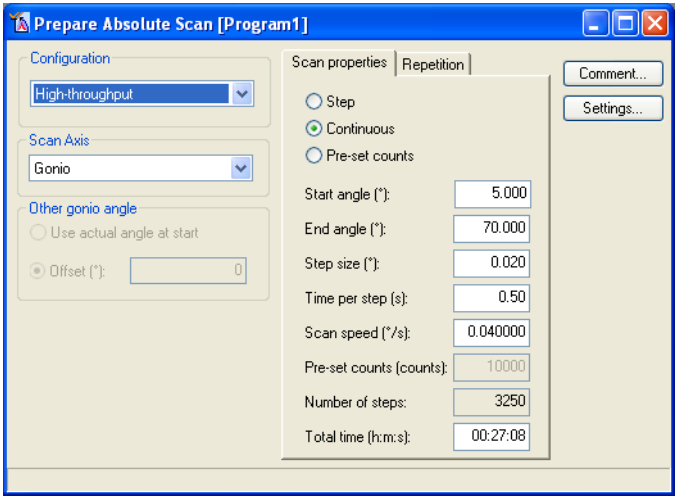
- Choose the type of program you want to define (in this example: "Absolute scan")



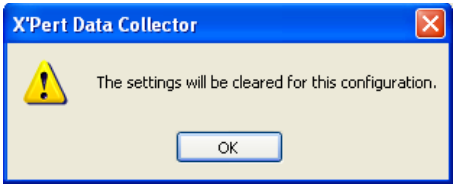
  and press OK.

3.5.1 Defining the Measuring Program Parameters

You have just opened the "Prepare Absolute Scan" window:



If the "Configuration" is not My Spinner, select it from the drop-down list. This will cause the following message to be displayed:



- Press OK.
- In the "Configuration" frame, if applicable, select:

  Diffracted Beam Path (these radio buttons only appear if your system has a double detector arm), choose the beam path with Bragg-Brentano focussing optics

- In the "Scan Axis" frame, select:

  Gonio (default)

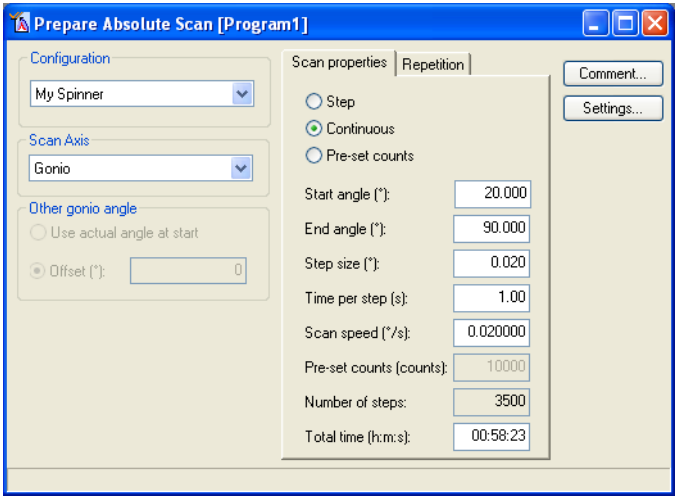
- In the "Scan Properties" tab, select:

	Continuous (default)
Start angle (°)	20
End angle (°)	90
Step size (°)	0.020 (default)

NOTE: If you have a an X'Celerator detector or a Position Sensitive Detector (PSD) in your system the step size is determined by the system according to the parameters of an X'Celerator or the PSD (defined when you press the Settings... button).

Time per step (s) 1

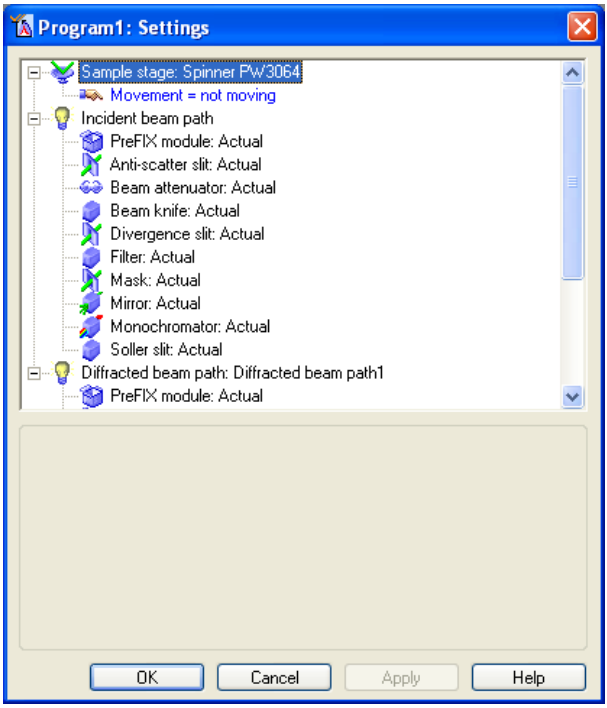
The Scan speed and Total time are automatically calculated when you leave this field.



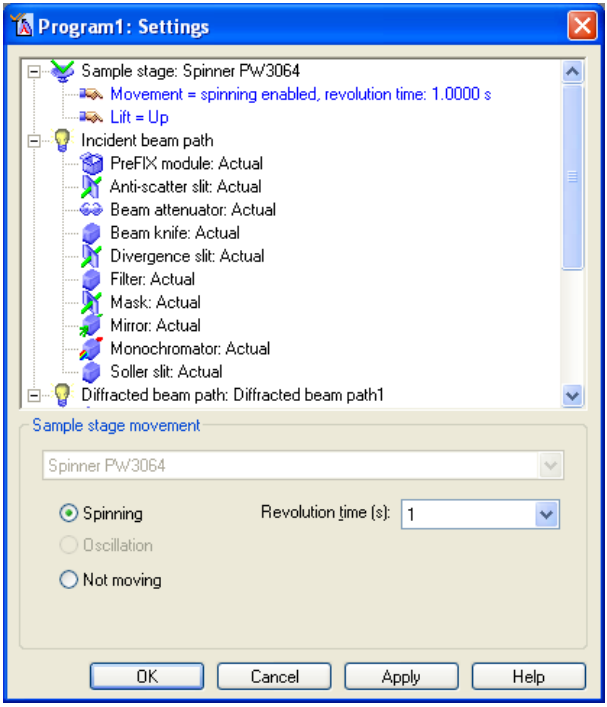
The next step is to define the hardware settings for the measurement that we have just defined.

3.5.2 Defining the Instrument Settings for the Measurement

- Press Settings... to open a window in which we can specify the actual hardware settings that we are going to use.



- Now select the Stage and Optics settings for this program. Click on the Sample Stage icon, select "Spinning" and if appropriate: the "Revolution time (s):" of "1".



- In the "Incident beam path" branch of the tree we choose which of the items that we pre-selected that we want to use in this program, starting with the PreFIX module, in this example: Programmable Divergence Slit:

Divergence slit:	Prog. Div. Slit (see Note following)
Usage:	Automatic
Usage:	Automatic
Irradiated length (mm):	10
Offset (mm):	0

NOTE: If you do not have a programmable divergence slit available to you, either select a fixed slit of 1° or an automatic slit with irradiated length = 12 mm, whichever is available to you.

Mask:	Inc. Mask Fixed 10 mm (MPD/MRD)
Soller Slit:	Soller 0.04 rad.

- In the "Diffracted beam path" branch of the tree we choose which of the items that we pre-selected that we want to use in this program, starting with the PreFIX module:

Anti-scatter slit:	Prog. AS Slit
Usage:	Automatic
Irradiated length (mm):	10
Offset (mm):	0

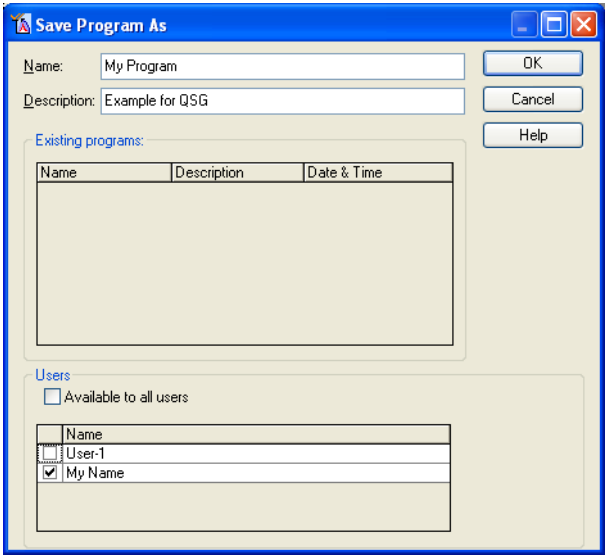
NOTE: If you do not have a programmable anti-scatter slit available to you, select a fixed AS slit of 1°.

Receiving slit:	Prog. Rec. Slit (with a height of 0.3).

NOTE: If you do not have a programmable receiving slit available to you, select a fixed receiving slit of 0.3 mm.

Soller slit:	Soller 0.04 rad.
Detector:	Select your detector.

- Press OK to apply the settings to your program. Complete the creation of the measurement program by saving it: *File – Save* (in this example: "My Program").

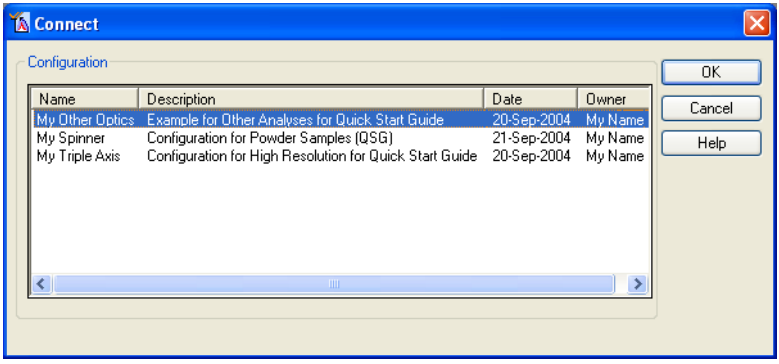


- Press OK and close the Prepare Absolute Scan window by pressing .

3.6 Performing the Measurement

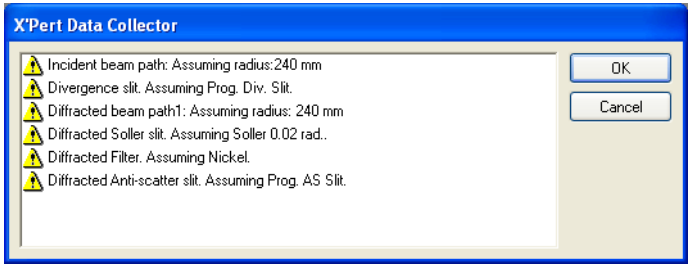
Before we can start a measurement we must go on-line (connect to the diffractometer):

- Select *Instrument - Connect.*

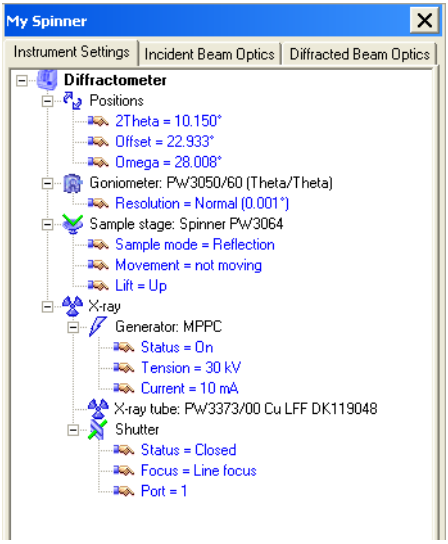


- Select the configuration that you made earlier (in this example: "My Spinner").
- If applicable - select the diffracted beam path with the Bragg-Brentano type optics and press OK.

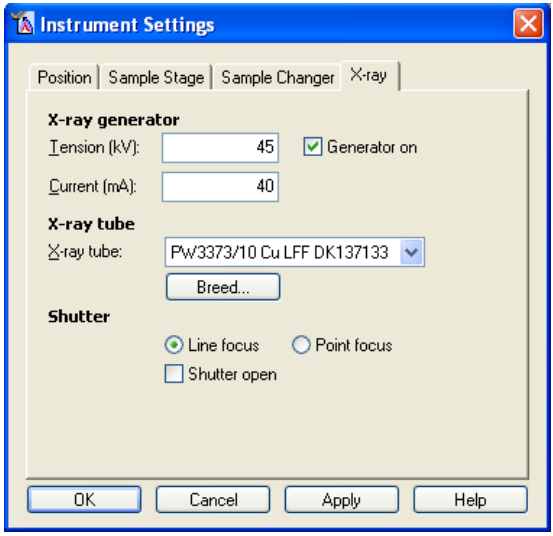
  A message window showing the 'assumed' status of the system is displayed:



- In order to make sure that you obtain a good measurement, you must carefully check these assumptions. If these assumptions are correct, press OK and proceed with the next step. If they are not correct you must still press OK and then go to the tab(s) on the instrument control window containing the incorrect assumption and make the corrections.
- Carefully look through the "Instrument Settings", "Incident Beam Optics" and "Diffracted Beam Optics" tabs to make sure that you have the correct parts mounted.



- In the "Instrument Settings" tab double-click on the "Generator" path of the tree and enter "45 kV" and "40 mA":



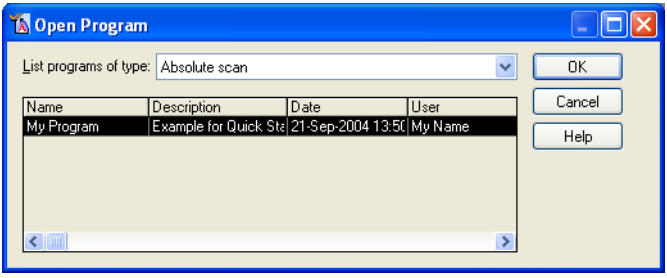
NOTE: If you have a PSD you should select a lower value (for example: "35 kV" and "30 mA") to avoid saturating the detector.

- Press Apply, and the system will power up to 45 kV and 40 mA.

- Depending on your situation, mount the Si sample as follows:

If you do not have a sample spinner in your system:	Mount the sample on the sample stage, close the enclosure doors and press OK.
If you do not have a sample changer in your system:	Select the "Sample Stage" tab. Use the handle to lower the sample spinner platform, mount the sample, release the handle to bring the sample to the spinning position. Close the enclosure doors and press OK.
If you have a sample changer, but it is positioned in the corner:	Select the "Sample Stage" tab. Uncheck "Lift Up", close the doors and press Apply. Open the doors, mount the sample, close the enclosure doors, check "Lift Up" and press OK.
If you have a sample changer, and it is ready to use:	Select the "Sample Changer" tab. Open the doors, put the sample into an empty magazine or monitor position. Close the doors, indicate to load the sample from the position you just loaded the sample into and press OK.

- Start the measurement program by selecting *Measure – Program…*



- Highlight your program (in this example: "My Program") and press OK.
- In the "File" frame enter:

  Name: My Program_1.xrdml (default)

  Folder: C:\X'Pert Data\My Name (default)

  Comment: (in this example: "Example for Quick Start

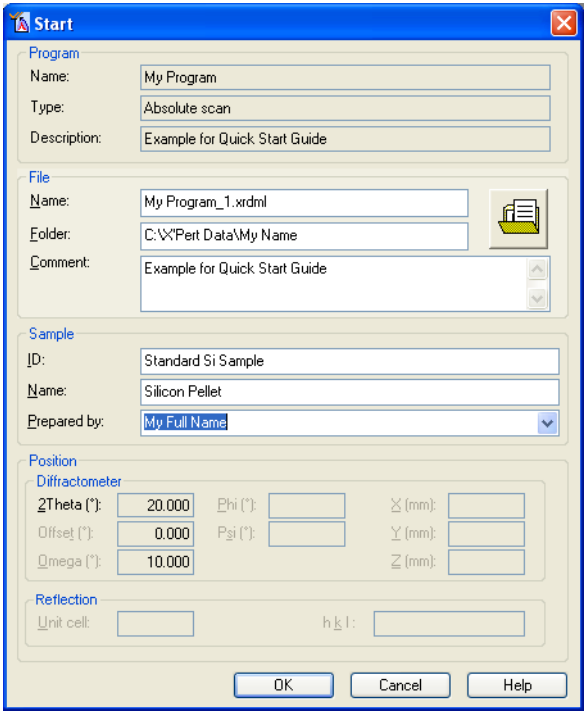
Guide").

- In the "Sample" frame, give the sample a name:

  ID: Standard Si Sample

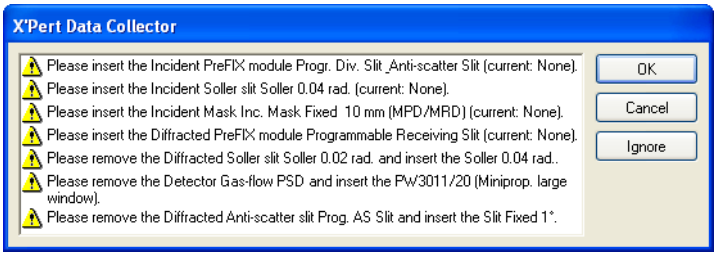
  Name: Silicon Pellet

  Prepared by: select "My Full Name" from the drop-down list ( ).



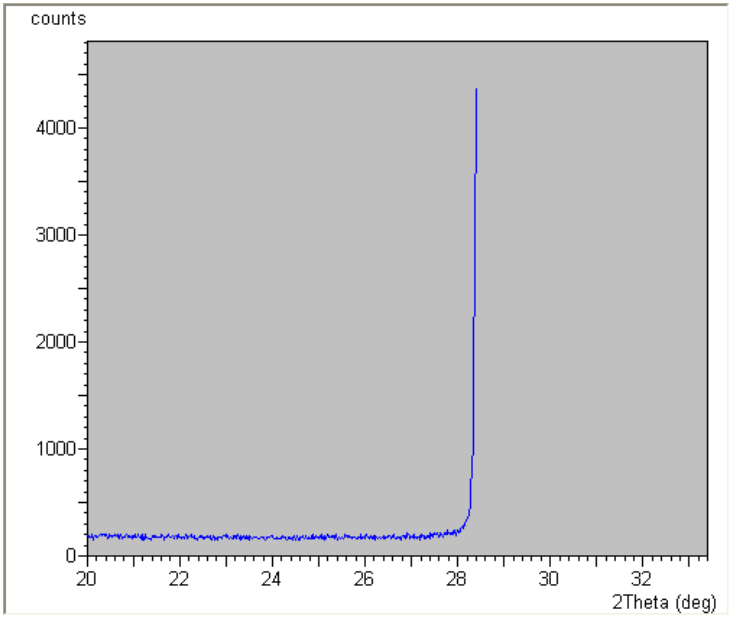
- Check that the enclosure doors are closed and press OK.

  If any physical actions must be performed before the system can run the program, a list of these actions will be displayed.

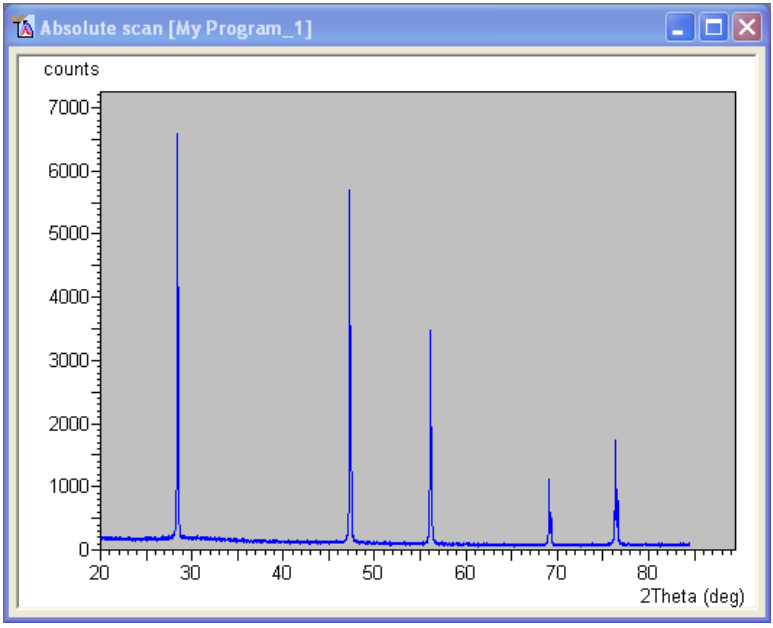


- If there are any actions to be done, perform them and then press OK.

  The scan starts and will take some time, depending on the program parameters (in this example: 1 hour). The scan is displayed as the measurement progresses:



  Notice that the scale changes as the measurement proceeds.



This scan is automatically saved with the file name "My Program_1.xrdml".

You have now collected the data.

You can now use X'Pert Data Viewer to view your results. A guide to using X'Pert Data Viewer is given in the X'Pert Explorer Add-ons Quick Start Guide (4022 339 07591).

If you want to automatically use the results of future measurements, you could for example, utilize one of the scripts available with X'Pert Automatic Processing Program. Examples of these scripts and of how to use them is given in the X'Pert Automatic Processing Program - Quick Start Guide (4022 339 07891).

- Close X'Pert Data Collector by selecting *File* - *Exit* and pressing OK.

X'Pert Data Collector - Quick Start Guide

PANalytical

Chapter 4

Using X'Pert Data Collector with MRD Type Systems

Contents

X'Pert Data Collector - Quick Start Guide

4.1 Introduction

This chapter describes how to use X'Pert Data Collector with a Materials Research Diffraction (MRD) type System (system with an MRD cradle and high resolution optics, or an MRD XL cradle and high resolution optics) to measure the silicon (111) single crystal sample delivered with the system (section 4.5 - Measuring the Rocking Curve).

It also describes how to use X'Pert Data Collector with a Materials Research Diffraction (MRD) System to measure a thin film sample at very shallow (small 2Theta) angles (section 4.6 - Performing a Reflectivity Measurement). For this part we assume that a beam attenuator is present in the system (as it should be).

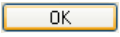
We presume that you have defined a user "My Name" as described in Chapter 2 of this guide.

4.2 Preparation

- Mount the sample on the sample stage.
- Switch the system on by pressing the "Power On" button on the diffractometer. When the kV display shows 30 and the mA display 10, the system is ready for use. If the power does not run to 30 kV and 10 mA please refer to the relevant hardware User's Guide.

4.3 Starting X'Pert Data Collector

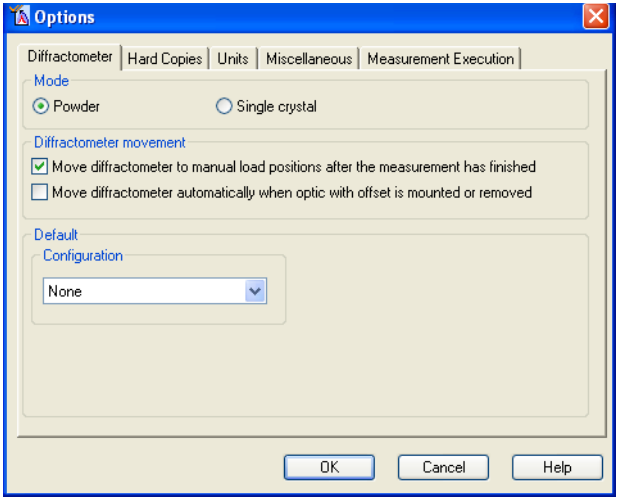
- Double-click on the X'Pert Data Collector icon 

.
- Enter the user name and password: "My Name" and "password" and press 

.

You should check that the system is set up to use the diffraction mode that you want, in this case, Single Crystal Mode:

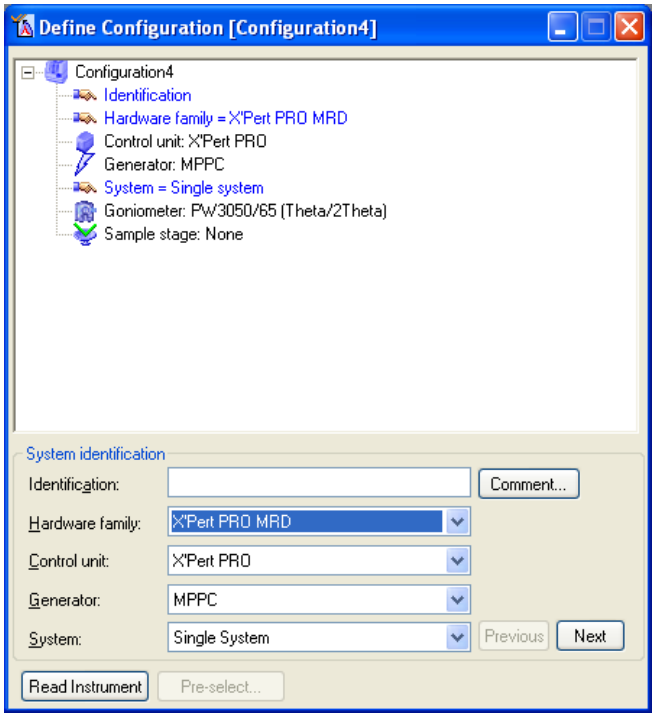
- Select *User Settings – Options*.



- Choose the mode: "Single crystal" and press OK.

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

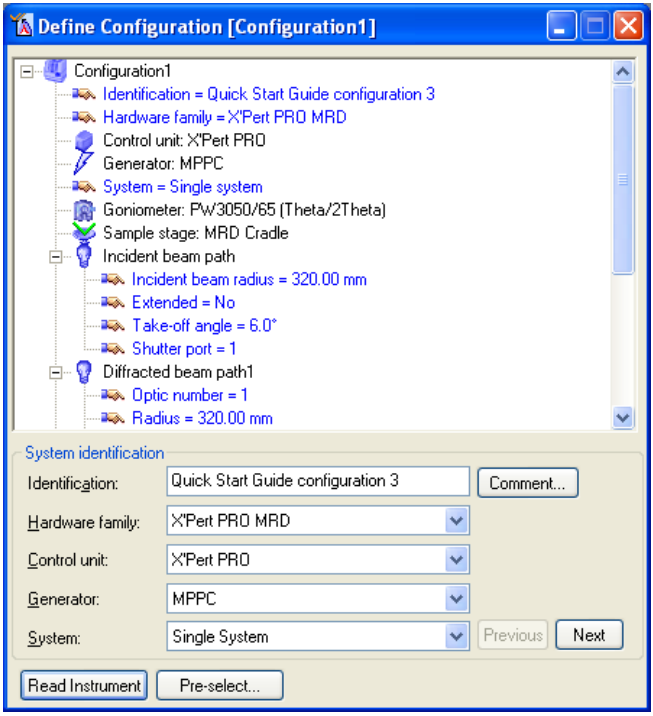
- Select *File – New Configuration...*
- Select the "Hardware family" (in this example: "X'Pert PRO MRD")



and then press Read Instrument.

If the system has not been previously initialized an initialization wizard will start. This wizard is designed to handle situations where there is a possibility of collision when the diffractometer resets (for example: system parts might collide). If this wizard does start, just follow the instructions displayed on the screen.

If the system cannot uniquely identify some of the items it will ask you to specify what you have.



The next step is to set up two new configurations one with two beam paths, and the other with one beam path; in other words tell the computer what the system "looks" like.

4.4 Setting Up Two Configurations

In this example we have a diffractometer with a requirement for two configurations:

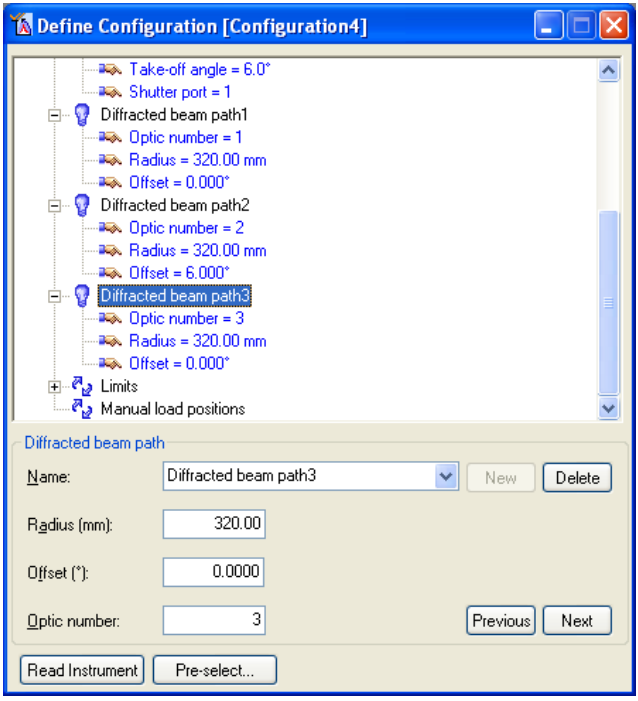
1. A rocking curve - triple axis configuration with two detectors.
2. A configuration containing the remaining diffracted beam optics.

4.4.1 First Configuration; Rocking Curve – Triple Axis

Removing the unnecessary beam path

If you have an MRD type system with diffracted beam optics other than rocking curve - triple axis PreFIX module you will see an extra diffracted beam path. Diffracted beam path 1 and Diffracted beam path 3 are mutually exclusive as both of them have an offset of 0.000º. In this example we will remove Diffracted beam path 3.

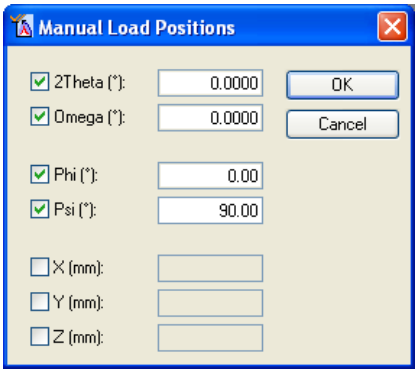
- Click on the Diffracted beam path that you are going to delete (scroll down if necessary). In this example "Diffracted beam path3".



- Press Delete.

Defining the rest positions

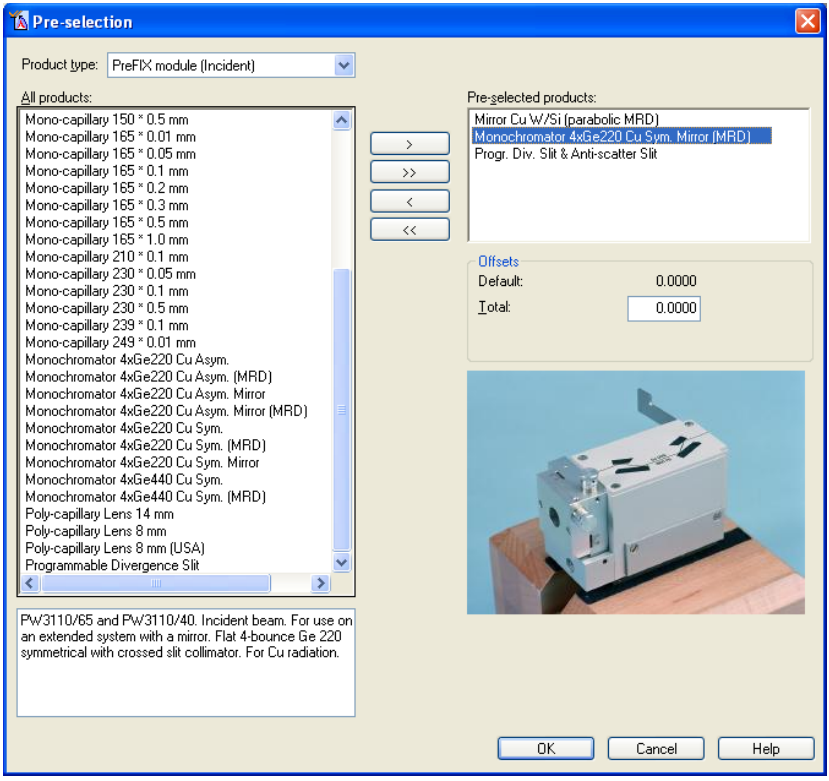
- Click on the "Manual load positions" branch of the tree and then on Manual Load Positions... . Set the check boxes as required:



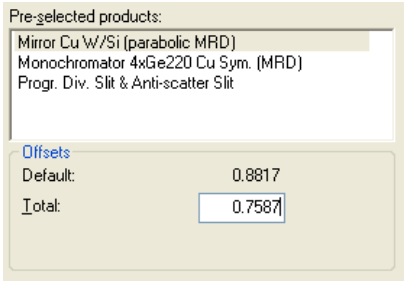
- Press OK .

Selecting your optical components

- Press the button Pre-select... to obtain a series of pages where you can tell the system what changeable devices are available to you. Always start with the PreFIX module products (default is "PreFIX module (Incident)"), then "PreFIX module (Diffracted)" if any are available.

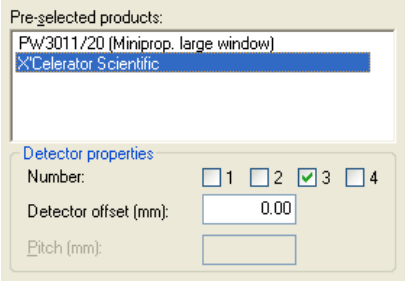


If you have X-ray Mirror or Hybrid monochromator PreFIX modules in your system it is important that you enter their offsets that are provided in the System Acceptance Form delivered with your system.

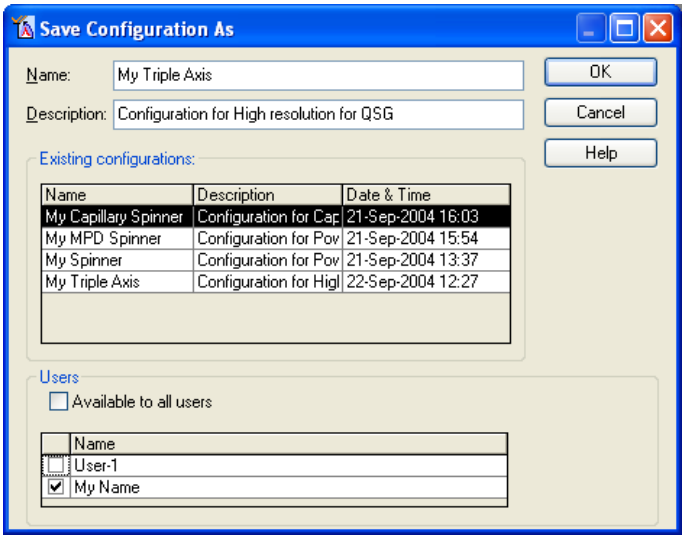


- Leaf through each entry in the "Product type" drop-down list and select those products that are available to you, and that you want to use in this configuration. To select an item, you highlight that item in the "All products" list and then press [>].

  If your system includes an X'Celerator detector (and an X'Celerator detector monochromator) or a position sensitive detector it is important to define the detector offset provided in the System Acceptance Form delivered with your system.



- When you have selected all of the items that you require press [OK].
- Save this configuration by selecting *File – Save*. Enter the name of the configuration, in this example: "My Triple Axis".



- Now press OK and then close the "Define Configuration" window by clicking .

If you have other optics in your system proceed with the following section (4.4.2 "Second Configuration; Other Optics"), if not skip that section and go directly to section 4.5 "Measuring the Rocking Curve".

4.4.2 Second Configuration; Other Optics

Now we will set-up the 2nd configuration with the beam path carrying the other optics.

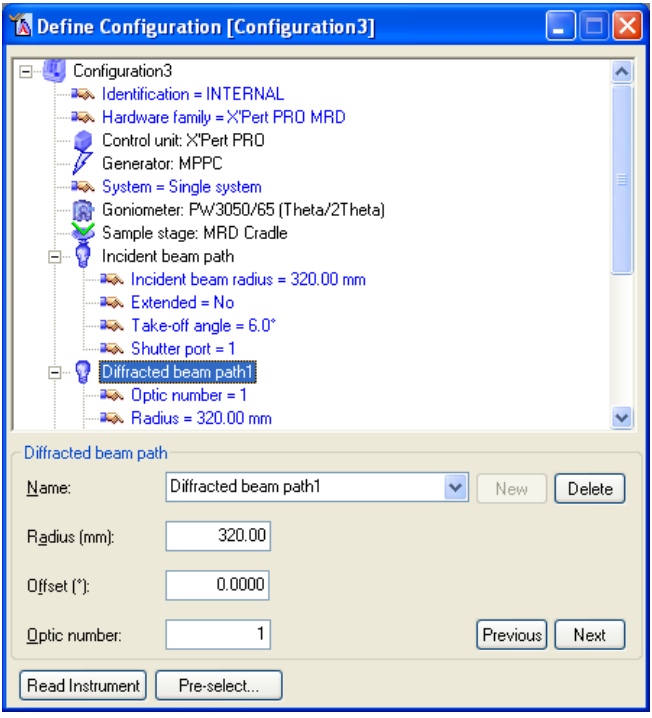
- Select *File – New Configuration…*
- Select your Hardware family (in this example: "X'Pert PRO MRD") from the drop-down list and press Read Instrument to obtain the instrument configuration.

  If the system has not been previously initialized an initialization wizard will start. This wizard is designed to handle situations where there is a possibility of collision when the diffractometer resets (for example: system parts might collide). If this wizard does start, just follow the instructions displayed on the screen.

If the system cannot uniquely identify some of the items it will ask you to specify what you have.

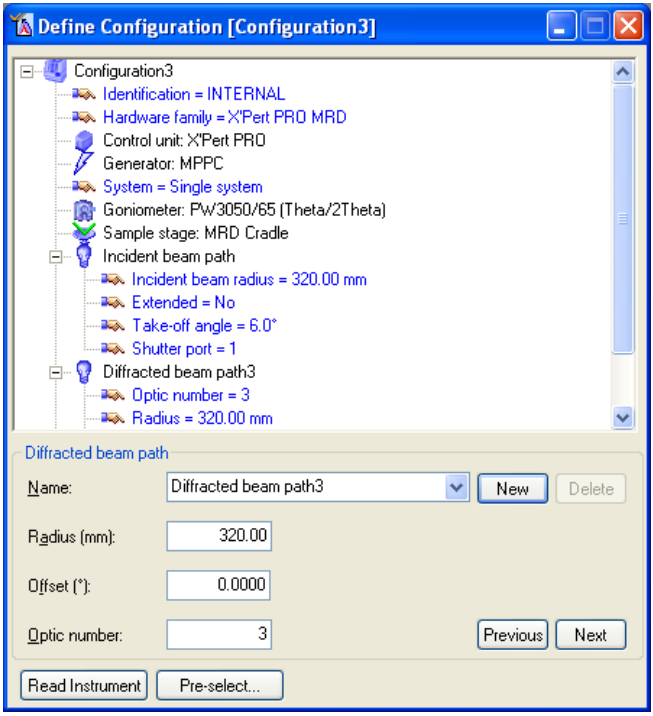
As this configuration only uses the beam path that we removed in section 4.4.1 we must now remove the other two beam paths from the configuration (in this example: "Diffracted beam path1" and "Diffracted beam path2").

- Select "Diffracted beam path1"



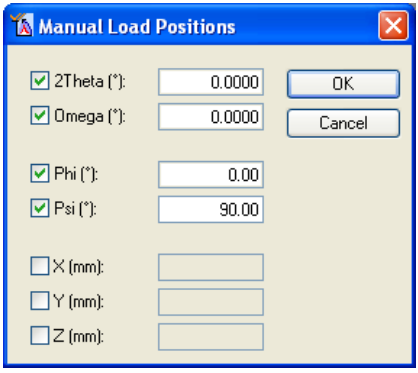
and remove it from the configuration by pressing Delete.

- Select "Diffracted beam path2" and remove it from the configuration by pressing Delete.

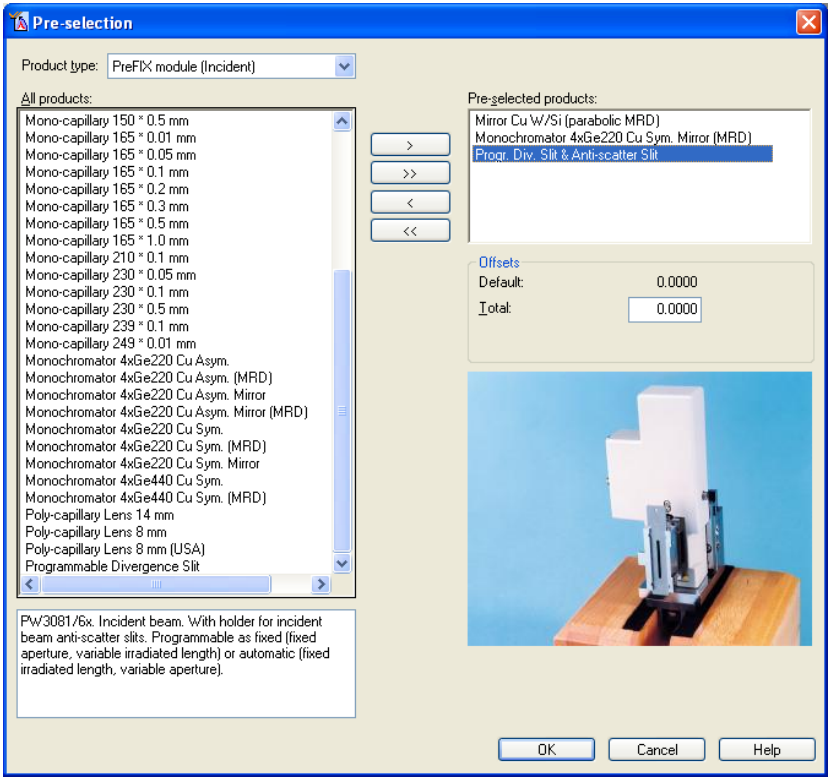


Define the manual loading positions:

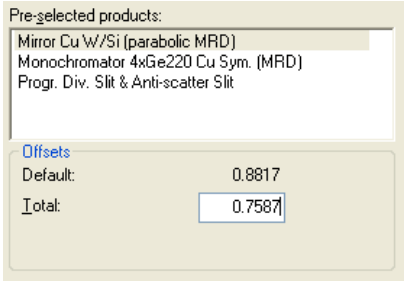
- Click on "Manual load positions" branch of the tree and then on Manual Load Positions... . Then set the check boxes as required:



- Press OK.
- Press the button Pre-select... to obtain a series of pages where you can tell the system what changeable devices are available to you. Always start with the PreFIX module products (default is "PreFIX module (Incident)"), then "PreFIX module (Diffracted)" if any are available.

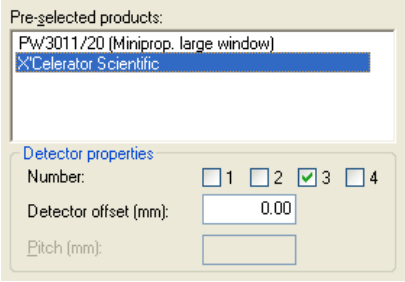


If you have X-ray Mirror or Hybrid monochromator PreFIX modules in your system it is important that you enter their offsets that are provided in the System Acceptance Form delivered with your system.

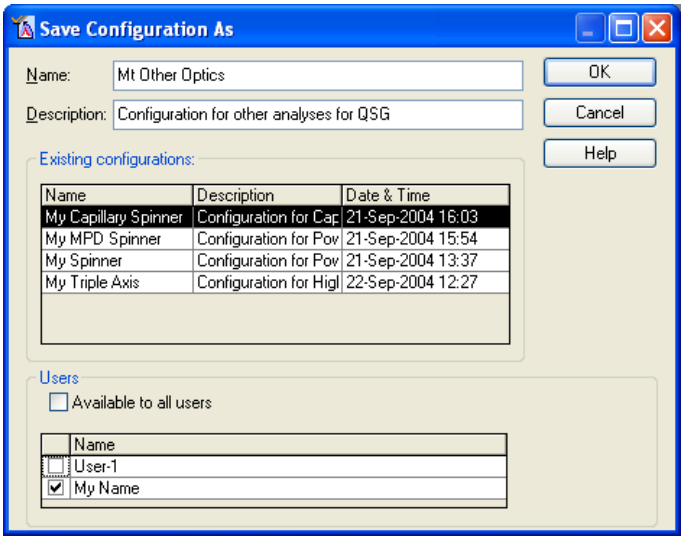


- Leaf through each entry in the "Product type" drop-down list and select those products that are available to you, and that you want to use in this configuration. To select an item, you highlight that item in the "All products" list and then press [>].

  If your system includes an X'Celerator detector (and an X'Celerator detector monochromator) or a position sensitive detector it is important to define the detector offset provided in the System Acceptance Form delivered with your system.



- When you have selected all of the items that you require press [OK].
- Save this configuration *File – Save* (or - *Save As*):
- Enter the name of the configuration, in this example: "My Other Optics", and the description "Configuration for other analyses for QSG".



and press OK.

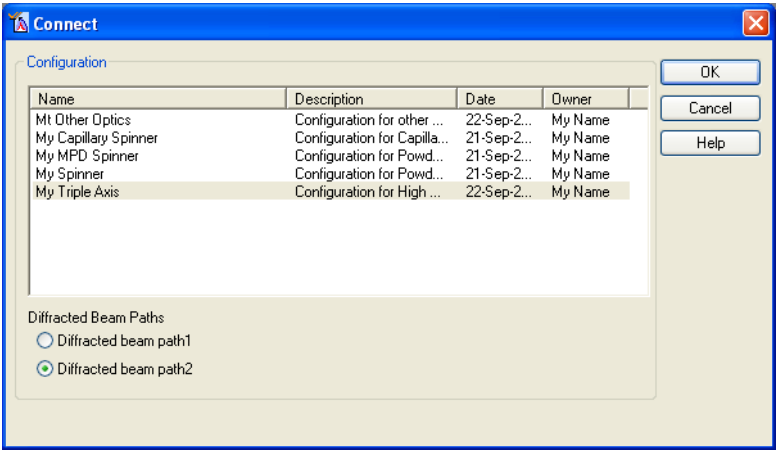
- Close the "Define Configuration" window by clicking .

4.5 Measuring the Rocking Curve

Now that we have prepared the configurations we can proceed with the measurements.

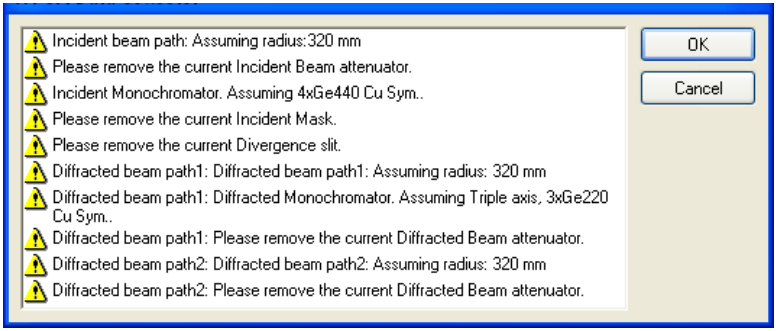
4.5.1 Going On-line

- Select *Instrument - Connect.*
- Select your configuration (in this example: "My Triple Axis").
- Select the beam path (in this example: "Diffracted beam path2")



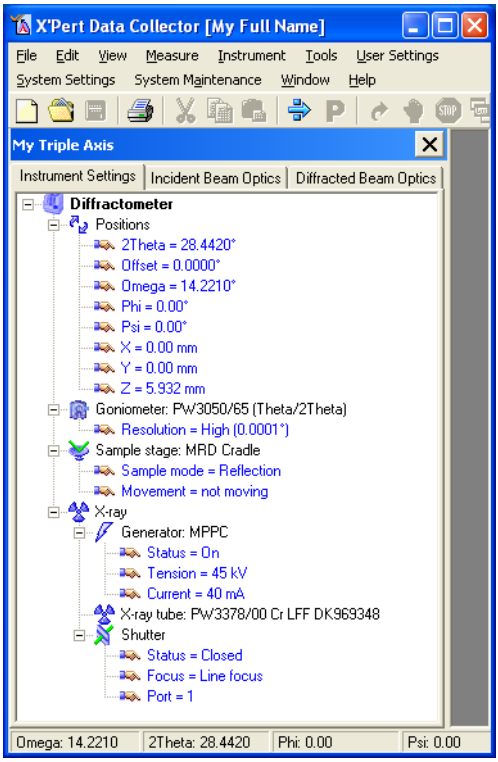
and press OK.

You may see a message window telling you the assumptions that the software has made about the status of the system.

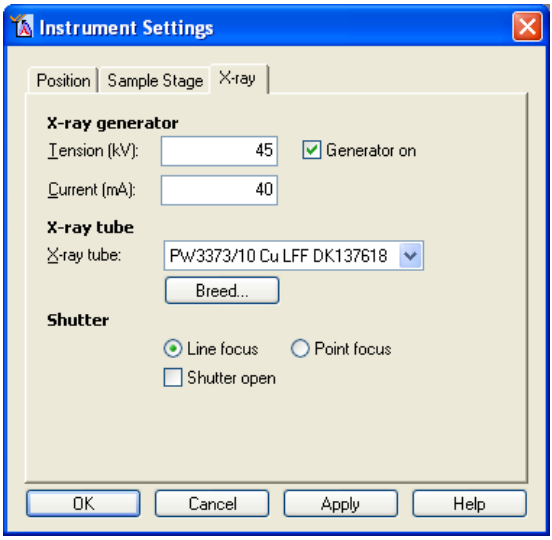


In order to make sure that you obtain a good measurement, you must carefully check these assumptions. If these assumptions are correct, press OK and proceed with the next step. If they are not correct you must still press OK and then go to the tab and the branch on the instrument control window containing the incorrect assumption and make the corrections.

If you have an In-plane type system and it has not been initialized, you will get a message telling you so. If you do get this message, follow the procedure given in order to initialize the system.



- On the "Instrument Settings" tab, double-click on the "Generator" branch to open the Instrument Settings window at the "X-ray" tab. Enter the required values (in this example: "45 kV" and "40 mA").

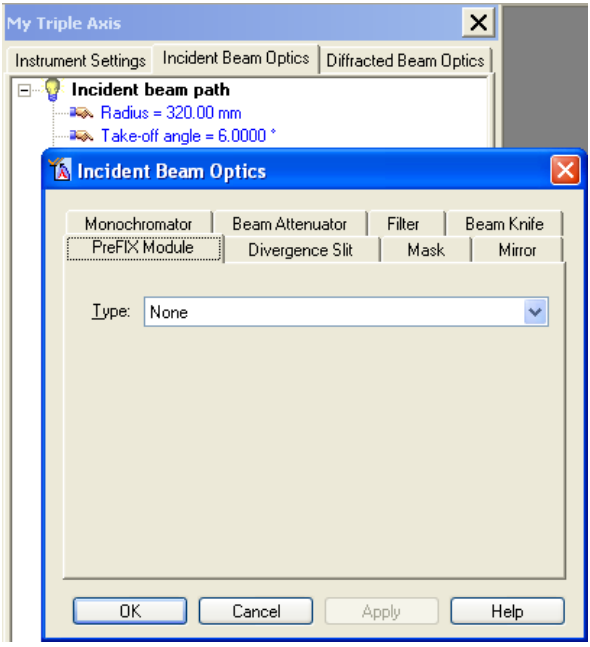

Instrument Settings
Position
Sample Stage
X-ray
X-ray generator
Tension (kV):
45
Generator on
Current (mA):
40
X-ray tube
X-ray tube:
PW3373/10 Cu LFF DK137618
Breed...
Shutter
Line focus
Point focus
Shutter open
OK
Cancel
Apply
Help

- Press OK.

4.5.2 Preparing the Beam Paths

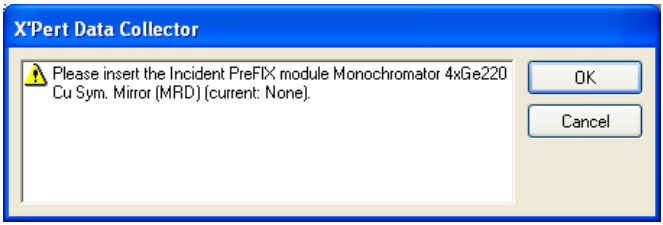
In this part of the example we will prepare and correct both the incident and diffracted beam paths. We will first prepare the incident beam path:

- Click on the "Incident Beam Optics" tab. Double-click on the "Incident beam path"

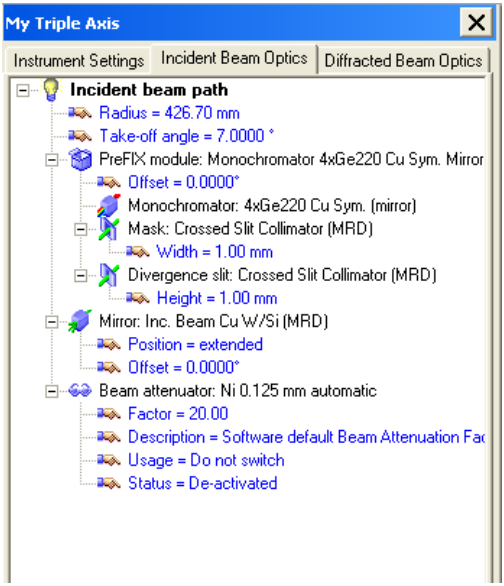


and then select the PreFIX module that you want to use from the drop-down list, press Apply.

Usually, at this time a window telling you to do something will appear.



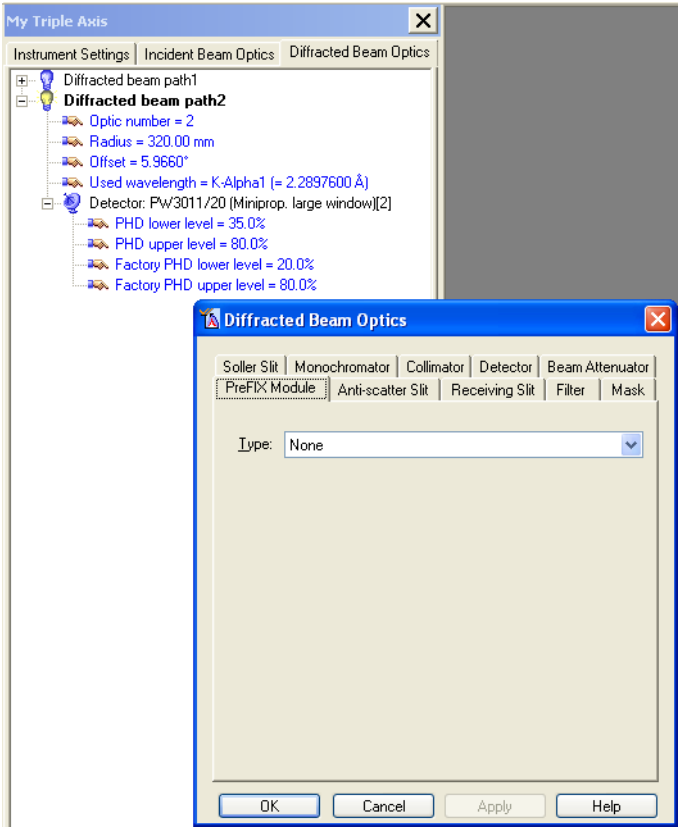
- Perform the actions as instructed and press OK.
- Carry on selecting the items in the beam path by selecting the relevant tabs and the types from the drop-down lists and pressing Apply.
- Press OK.



Now we will prepare the diffracted beam path:

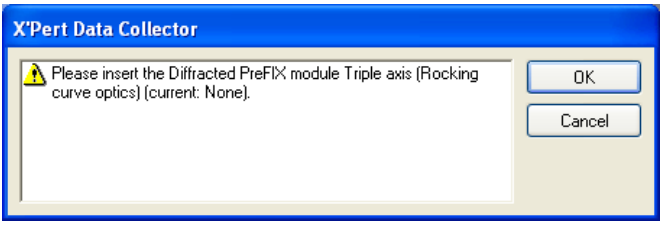
- Click on the "Diffracted Beam Optics" tab.

  You will see that the "Diffracted beam path2" is written in bold, indicating that it is the active beam path.
- Double-click on the "Diffracted beam path2".

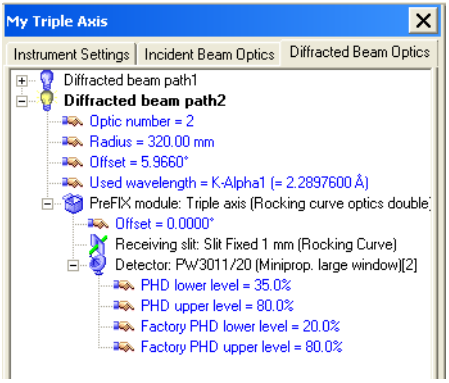


and then select the PreFIX module that you want to use from the drop-down list, press Apply.

Usually, at this time a window telling you to do something will appear.



- Perform the actions as instructed and press OK.
- Carry on selecting the items in the beam path by selecting the relevant tabs and the types from the drop-down lists and pressing Apply.
- Press OK.



4.5.3 Sample Positioning

Because we don't know exactly where the sample is mounted on the stage we must use the dial gauge to accurately pinpoint its position.

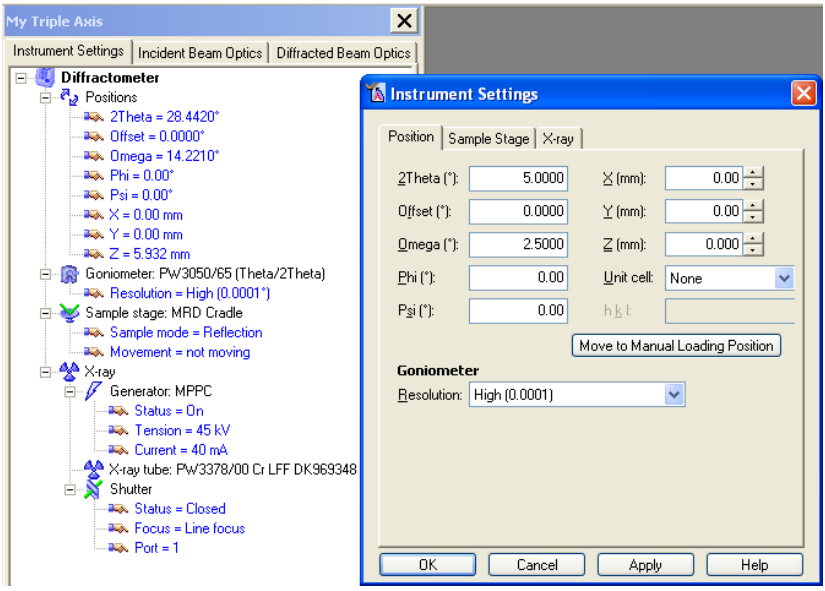
- If you haven't already done so, mount the sample (silicon 111 single crystal).
- Click on the "Instrument Settings" tab and double-click on "Positions".
- Position the sample stage so that you can read the dial gauge and not damage the sample (in this example):

  Psi : 0

  Z : 0.

- Press Apply.

  Mount the Z-position dial gauge (to be used as a pointer to the middle of the sample). Refer to the relevant hardware User's Guide for dial gauge mounting information. Vary the X (mm) and Y (mm) settings until the pointer is over the center of the sample.

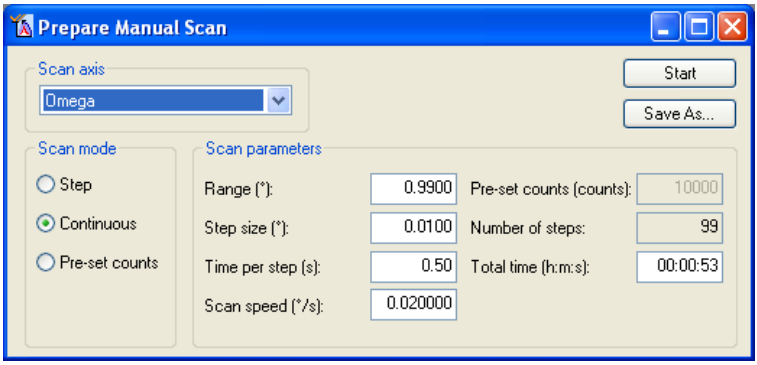


- To change the positions: enter the new value and press Apply.
- Carefully change the Z (mm) position until the sample is located exactly at the center of the goniometer (0 on the big scale on the dial and 1 on the small scale). Note the new Z position (in this example it was "7.226"). Change the Z position to 0 to prevent damaging the sample and dismount the dial gauge.
- Set the sample stage to a vertical position (Psi = 0°), set the Z position to the value just noted (7.226) and press OK.

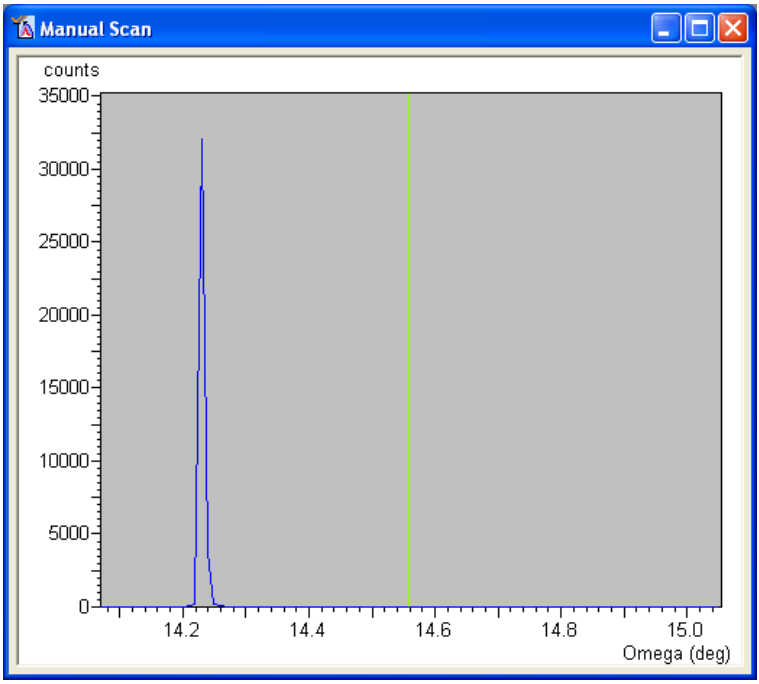
4.5.4 Manual Scan

Now we'll do a manual scan to "find" a reflected X-ray beam.

- Select *Measure – Manual Scan*
- Select "Omega" for the "Scan axis" and "Continuous" for the "Scan Mode".



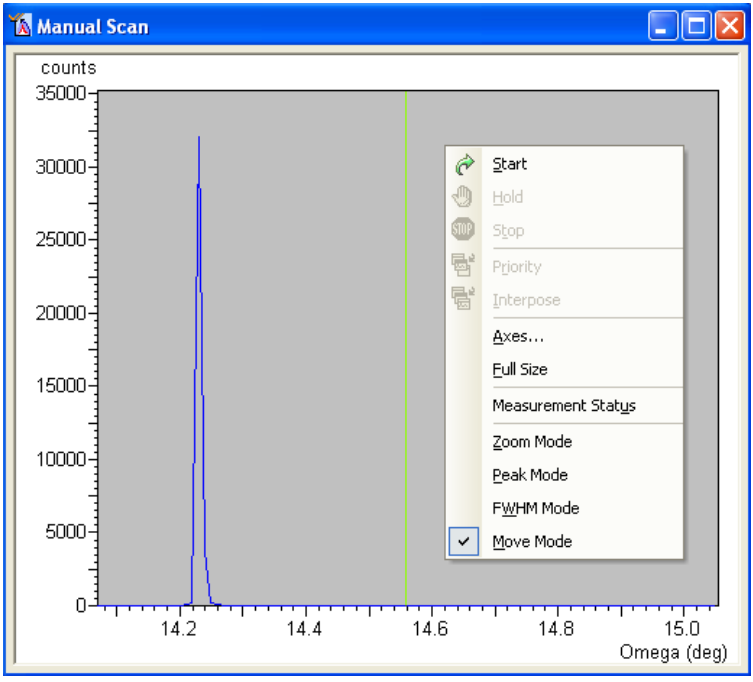
- In the instrument control window "Instrument Settings" tab, double-click on "Positions".
- Choose Si[111] for the "Unit cell" and 1 1 1 for "h k l" (be sure to enter this as 1, space, 1, space, 1), the position information will be automatically calculated and entered when you leave this field.
- Press OK.
- Press the Start button in the "Prepare Manual Scan" window.



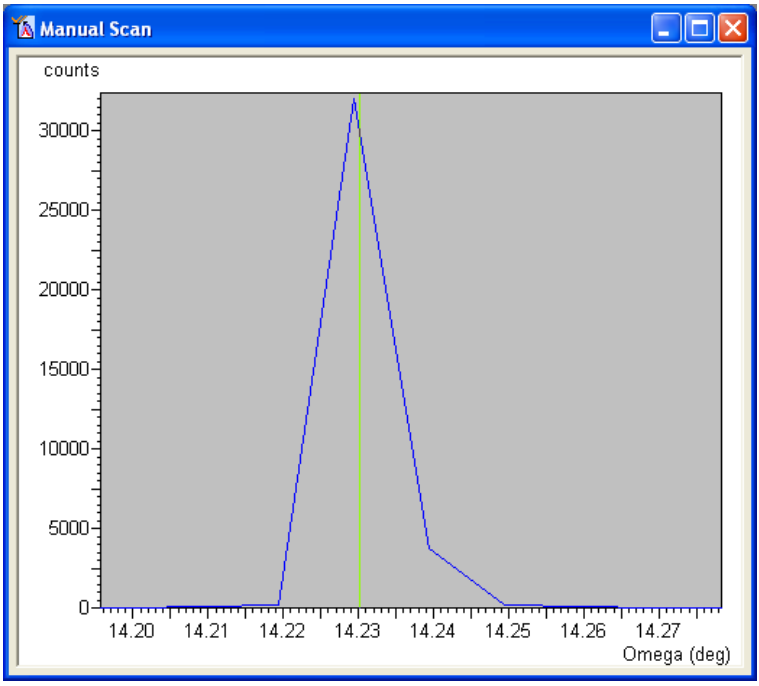
The result (count rate) varies considerably according to individual systems.

4.5.5 Adjusting the Goniometer Position

- Place your mouse pointer somewhere in the scan and press the right mouse button.
- Select *Move Mode* with the left mouse button.



- Use the left mouse button to "grab" the hairline, drag it to the center of the peak and release it. The goniometer moves to that angle.
- Once again use the right mouse button to get the pop-up list, select *Zoom Mode*, zoom in and look at the result:



If necessary, go back to Move mode and move the hairline into the middle of the peak.

- Press to close the "Manual Scan" window and the "Prepare Manual Scan" window.

4.5.6 Optimize Psi

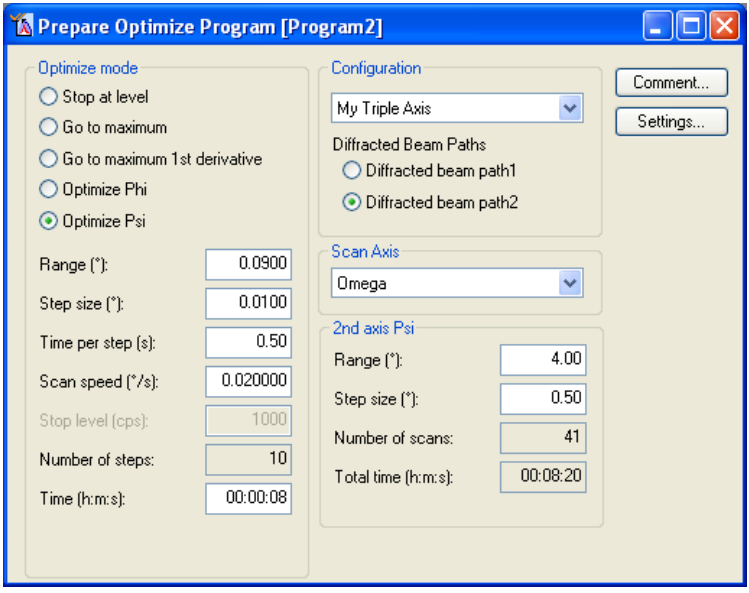
4.5.6.1 Prepare Optimize Program

- Select *File - New Program...*
- Choose the program type "Optimize Program" from the drop-down list.
- Press OK.
- Select "Optimize Psi".
- Set the following parameters:

Range:	0.090
Step size:	0.01
Time per step:	0.5

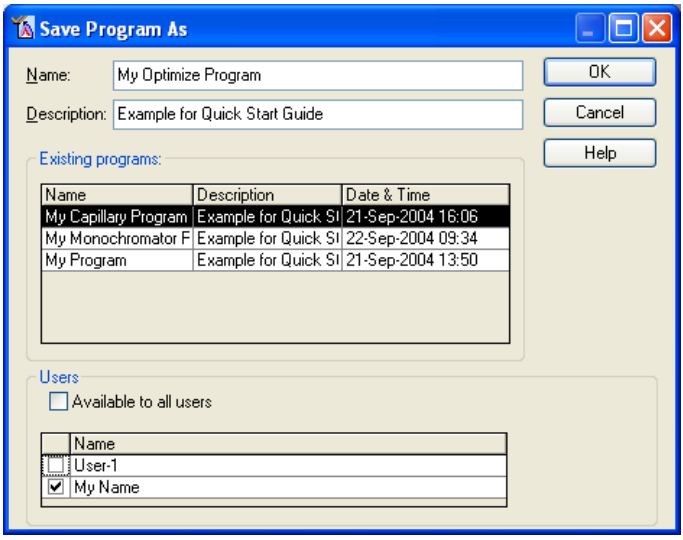
- For the 2nd axis, which is the Psi axis, set:

Range:	4
Step size:	0.5



- Select *File - Save*.
- Enter the name of the optimize program (in this example: "My Optimize Program") and the description "Example for Quick Start Guide".

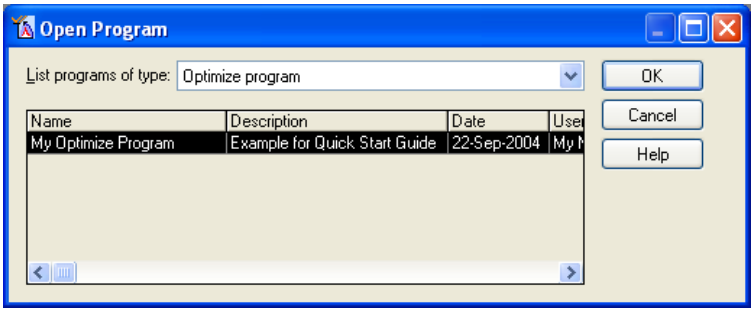
  Ensure that the user "My Name" is checked.



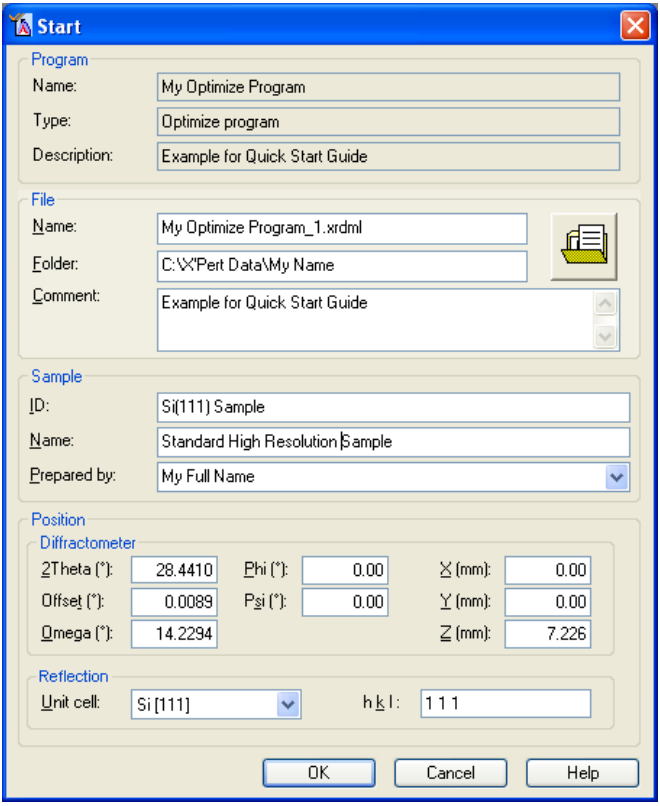
- Press OK and close the "Prepare Optimize Program" window ( ).

4.5.6.2 Optimizing the Sample Orientation

- Select *Measure - Program...*



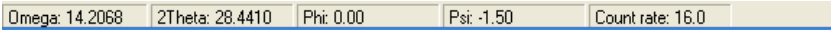
- Press OK.
- Either accept the proposed Name, Folder and Comment or change them to your requirements. Enter the Sample ID as "Si(111) Sample", the Sample name as "Standard High Resolution Sample", and select "My Full Name" from the "Prepared" by drop-down list.



- Press OK.

The sample orientation is now automatically optimized. This is done by performing a series of Omega scans at various fixed Psi settings.

After all the scans are finished the cradle automatically moves to the optimal Psi position and the shutter will be closed (the shutter number on the control console is no longer displayed). The optimized Psi position can be seen on the instrument control window on the "Instrument Settings" tab, and on the status bar. Use *User Settings - Status Bar* to select the items you want to display.



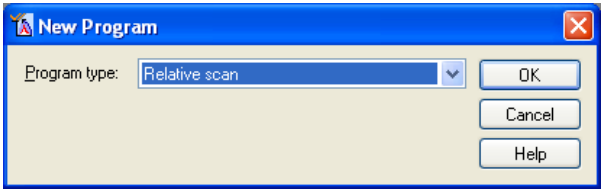
- Press to close the "Scan" window.

4.5.7 Measuring

We will now set up and execute the measurement program for a rocking curve.

4.5.7.1 Prepare Rocking Curve Program

- Select *File - New Program.*
- Choose "Relative scan" from the "Program type" drop-down list.

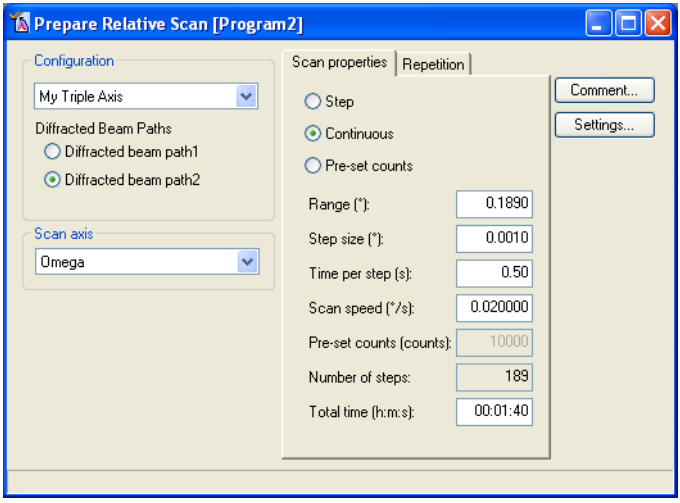


- Press OK.
- Check that you have the correct configuration and diffracted beam path (in this example: "My Triple Axis" and "Diffracted beam path2").
- Choose "Omega" for the scan axis and enter the following parameters:

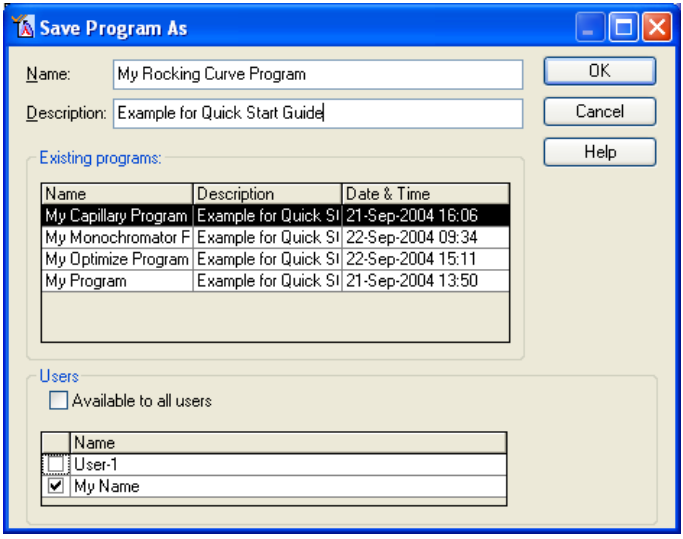
  Range: 0.189

  Step: 0.001

  Time per step: 0.5



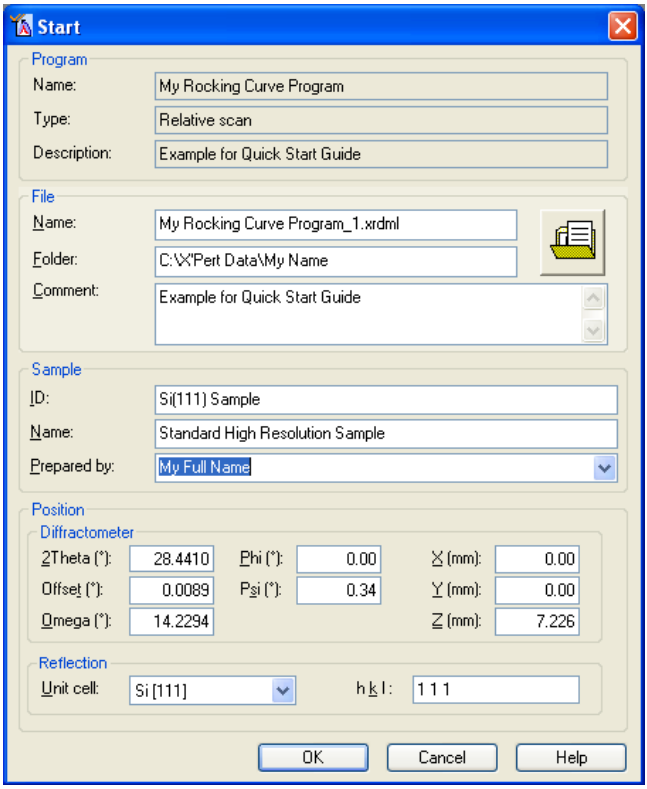
- Select *File – Save.*
- Enter the name of this measurement program (in this example: "My Rocking Curve Program").



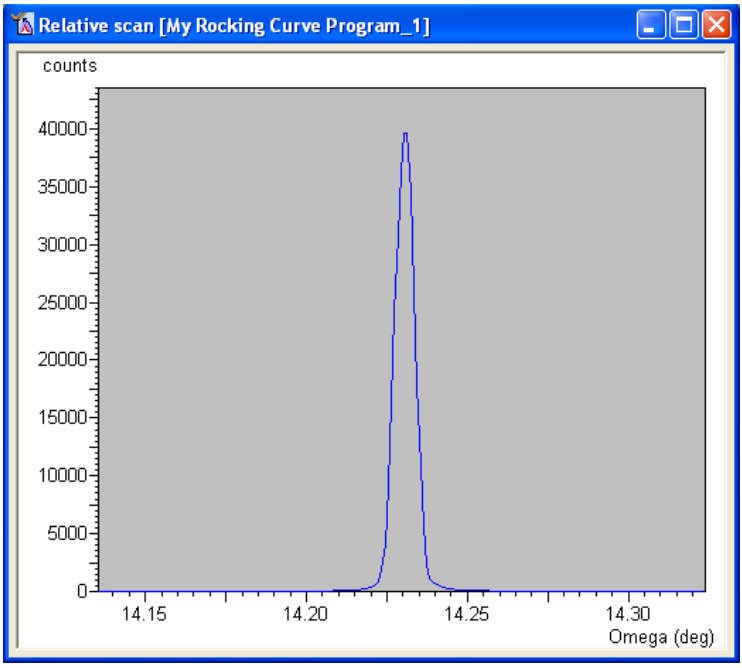
- Press OK.
- Close the "Prepare Relative Scan" window ( ).

4.5.7.2 Measure the Rocking Curve

- Select *Measure - Program...*
- Select "Relative scan" and "My Rocking Curve Program" and then press OK.
- Either accept the proposed Name, Folder and Comment or change them to your requirements. Enter the Sample ID as "Si(111) Sample", the Sample name as "Standard High Resolution Sample", and select "My Full Name" from the "Prepared by" drop-down list.



- Press OK. After a while the scan window will appear and shortly afterwards, your rocking curve measurement will be completed, at which time the goniometer will return to its manual load positions.



You have now collected the data.

You can now use X'Pert Data Viewer to view your results. A guide to using X'Pert Data Viewer is given in the X'Pert Explorer Add-ons Quick Start Guide (4022 339 07591).

If you want to automatically use the results of future measurements, you could for example, utilize one of the scripts available with X'Pert Automatic Processing Program. Examples of these scripts and of how to use them is given in the X'Pert Automatic Processing Program - Quick Start Guide (4022 339 07891).

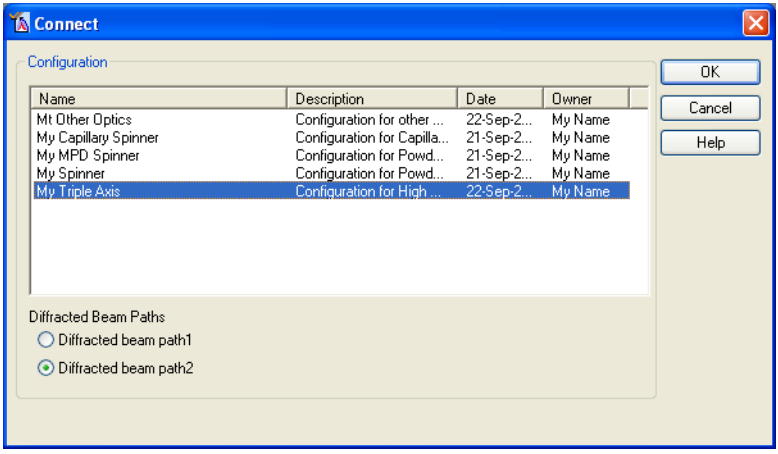
- Go off-line by selecting *Instrument - Disconnect* and then press OK.

4.6 Performing a Reflectivity Measurement

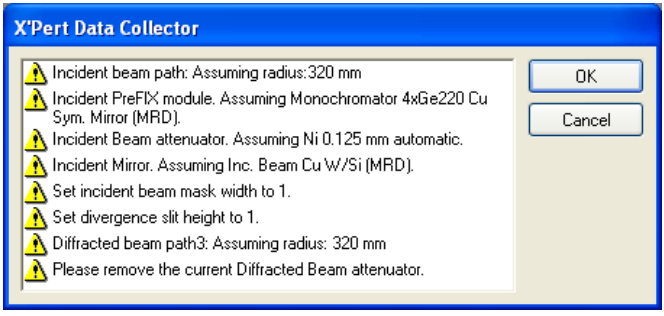
4.6.1 Going On-line

At this stage in the procedure you can either go on-line, or define a measurement program. In this example: go on-line.

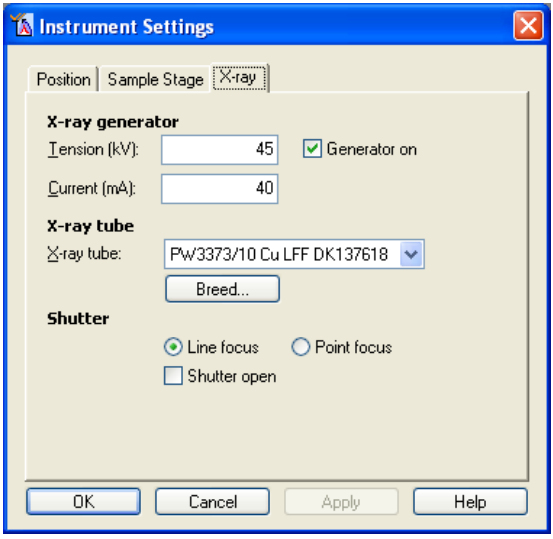
- Select *Instrument - Connect.*



- Select "My Other Optics" in the "Configuration" frame and press OK. A message window showing the "assumed" status of the system may be displayed:



- In order to make sure that you obtain a good measurement, you must carefully check these assumptions. If these assumptions are correct, press OK and proceed with the next step. If they are not correct you must still press OK and then go to the tab and the branch on the instrument control window containing the incorrect assumption and make the corrections.
- In the "Instrument Settings" tab set the X-ray tube parameters: double-click on "Generator" in the "X-ray" branch, and set the required values (in this example: "45 kV" and "40 mA") on the displayed window:

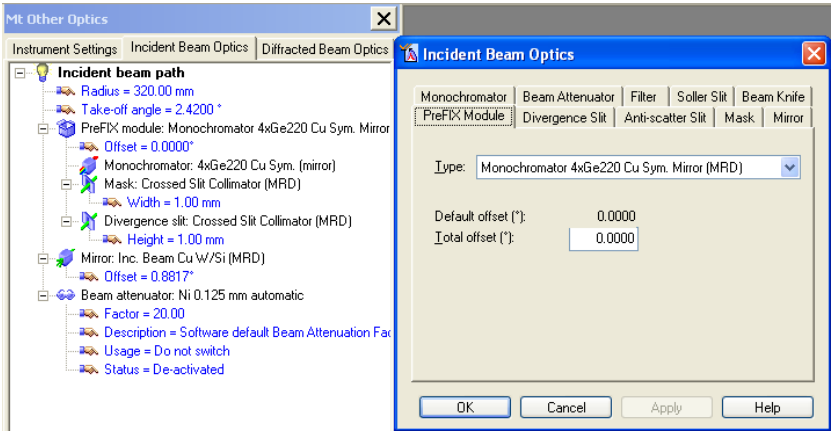


- Press Apply.
- When the system has powered up to 45 kV and 40 mA press OK.

4.6.2 Preparing the Beam Paths

In this part of the example we will prepare and correct both the incident and diffracted beam paths. We will first prepare the incident beam path:

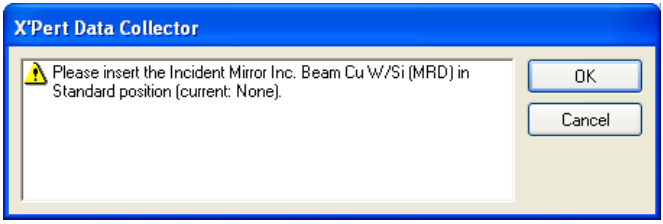
- Click on the "Incident Beam Optics" tab. Double-click on the "Incident beam path".

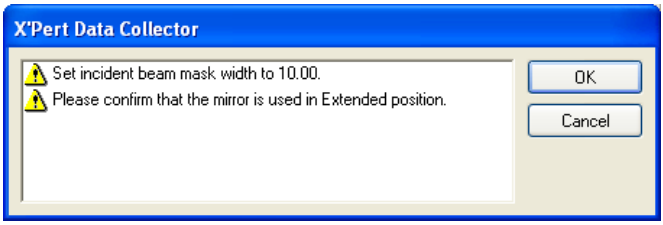


and then select the PreFIX module that you want to use from the drop-down list, press Apply.

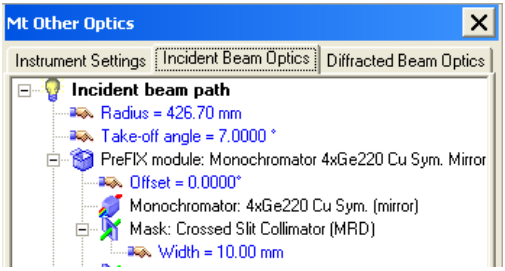
If you get a message telling you that you have actions to perform, do the actions as instructed and press OK.

- Select the items in the beam path by selecting the relevant tabs and the types from the drop-down lists.
- Press OK.





- Carry out the requested actions and then press OK. The incident beam optics reflect the actual situation.

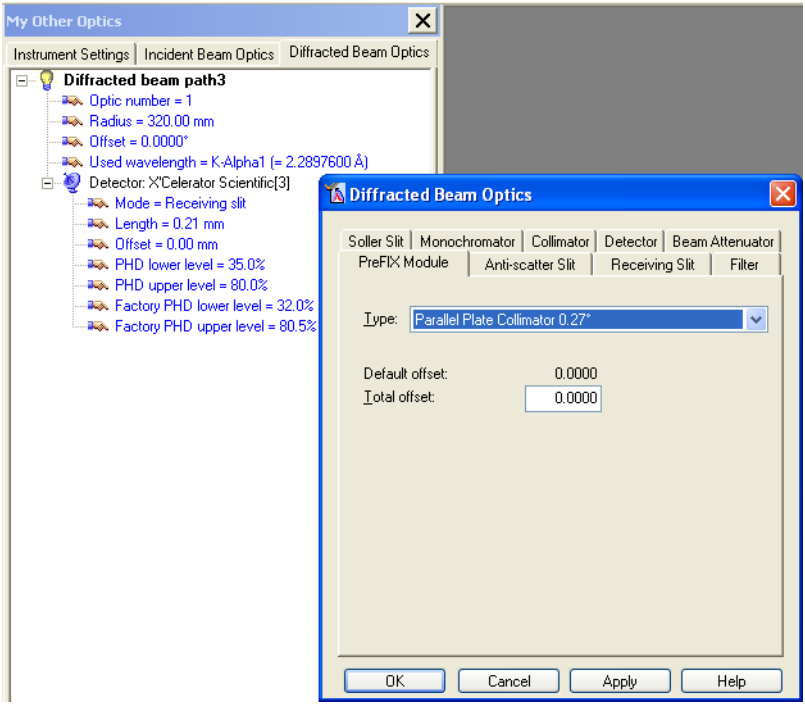


Now we will prepare the diffracted beam path:

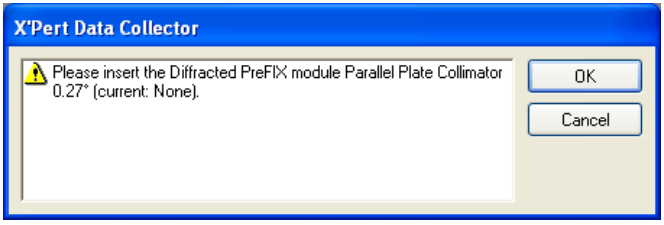
- Click on the "Diffracted Beam Optics" tab.

  You will see that the "Diffracted beam path3" is written in bold, indicating that it is the active beam path.

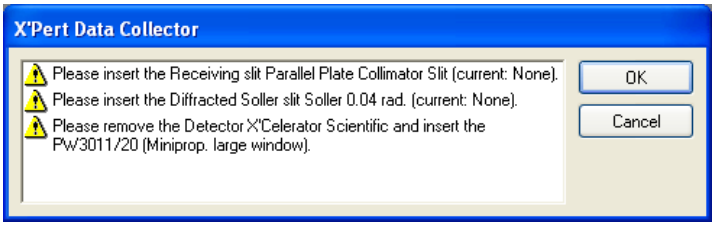
- Double-click on the "Diffracted beam path3" and then select the PreFIX module that you want to use for this parallel beam experiment from the drop-down list.



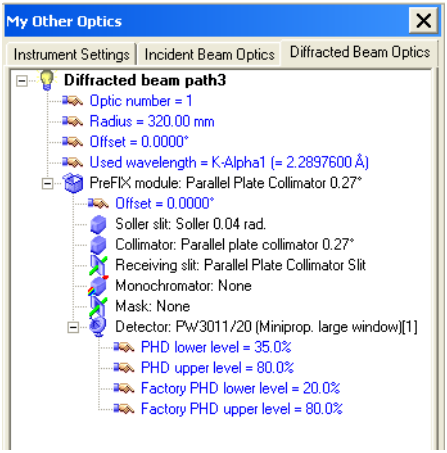
- Press Apply.



- Perform the actions as instructed and press OK.
- Select the items in the beam path by selecting the relevant tabs and the types from the drop-down lists.
- Press OK.

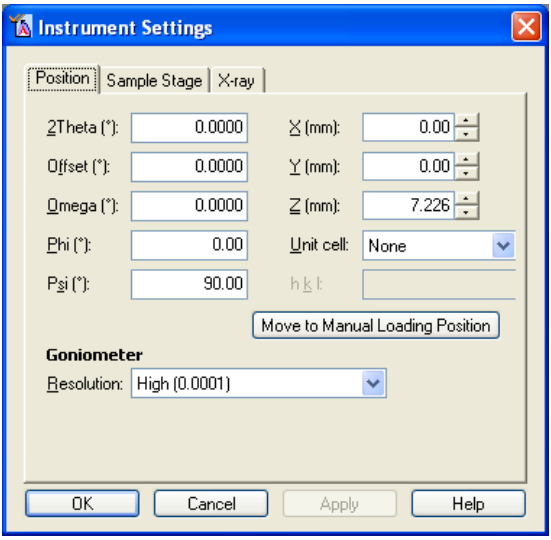


- Confirm that the required actions have been done by pressing OK.



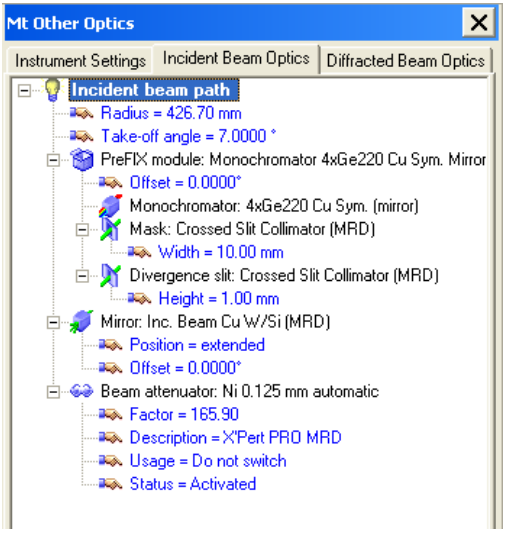
4.6.3 Mount the Sample

- Click on the "Instrument Settings" tab, then double-click on "Positions" in the tree.
- If required, send the goniometer to a comfortable position, for example by using the Move to Manual Loading Position button and mount the sample.



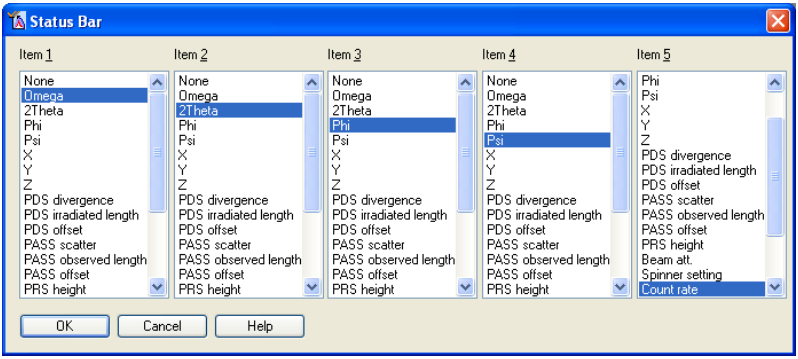
- Mount the Z-position dial gauge (to be used as a pointer to the middle of the sample), ensure that Z (mm) = 0 to avoid damaging the sample. Vary the X (mm) and Y (mm) settings until the pointer is over the center of the sample. To change the positions: enter the new value and press Apply.
- Adjust Z until the sample is at the correct height (refer to the relevant hardware User's Guide)
- Note the new Z position (in this example it was 7.984). Change the Z position to 0 to prevent damaging the sample and dismount the dial gauge.
- In order to ensure that the sample is at the precisely correct height we will first measure the output of the direct X-ray beam, and then move the sample up until the count rate is exactly half the previously measured level (= sample in the middle of the beam).
- Set: Psi to 0
  2Theta to 0
- Press Apply followed by OK.

- In the instrument control window select the "Incident Beam Optics" tab and double-click on the "Incident beam path".
- Choose the "Beam Attenuator" tab and then select "Do not switch" from the "Usage:" box drop-down list and tick "Activated".
- If you made any changes press Apply, regardless of whether or not you made any changes, press OK.

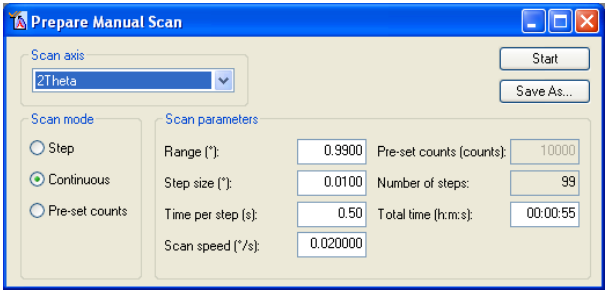


If you are using an uncalibrated beam attenuator you should calibrate the attenuation factor before you continue, if you have a calibrated one, go to section 4.6.4. An uncalibrated beam attenuator shows a beam attenuation factor of 20.00.

- Make sure that the status bar is switched on and shows the count rate. To do this select *User Settings* - *Status Bar* and check that "count rate" has been chosen for one of the five columns.

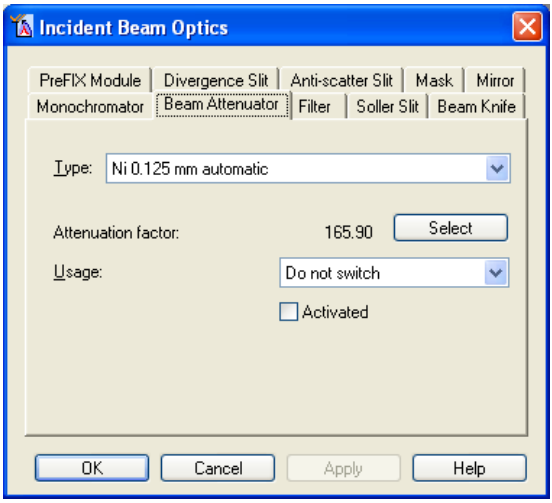


- Press OK.
- Select *View - Status Bar - Top* or *Bottom*.
- Select *Measure - Manual Scan.*
- Select "2Theta" from the "Scan Axis" drop-down list.

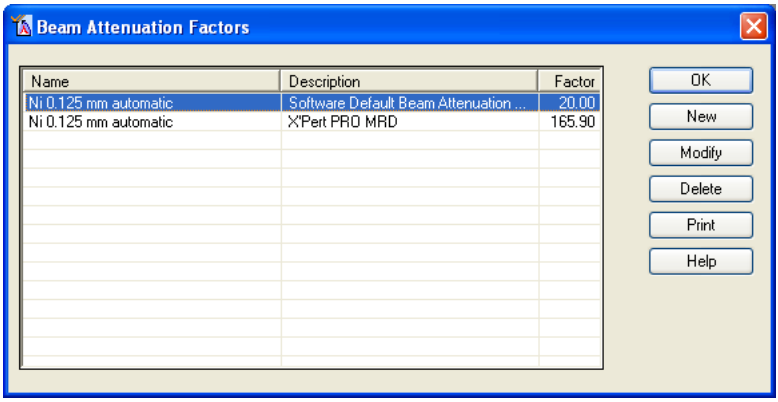


- Press Start.
- Check the count rate (**on the status bar**), if the count rate is above 1,000 go to the move mode (right-click in the graph) and move the hairline to a position on the peak where the count rate is below 1,000 (but preferably above 500).
- Close all of the manual scan windows by pressing on each of them.
- In the "Incident Beam Optics" tab of the instrument control window set the beam attenuator to 'Do not switch' and 'De-activated'.

- Press OK.
- Open the shutter by pressing the button on the toolbar.
- Double-click on the Beam Attenuator branch of the tree view.



- Take a note of the intensity shown in the status bar, activate the attenuator and press Apply. Then note the intensity again (in this example: "108000"). The attenuation factor is the second intensity divided by the first intensity.
- Set your beam attenuator to switch "At Pre-set Intensity", change the activate level to 500000 and press OK.
- Select *System Settings – Beam Attenuation Factors* press New and define the attenuation factor for your attenuator (in this example: "165.9").



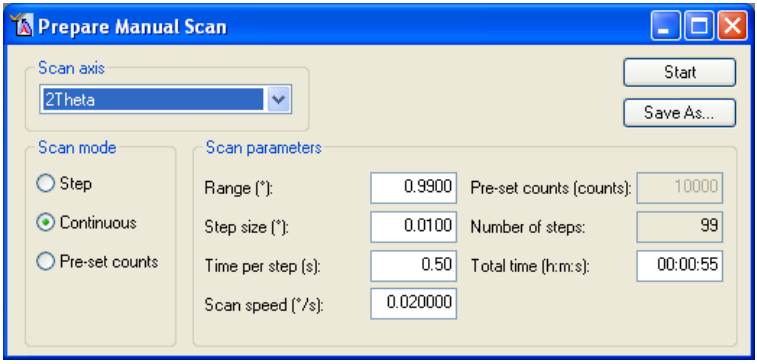
- Press OK.
- In the instrument control window select the "Instrument Settings" tab, double-click on the "Shutter" de-select "Shutter open" and press OK.

4.6.4 Manual Scan

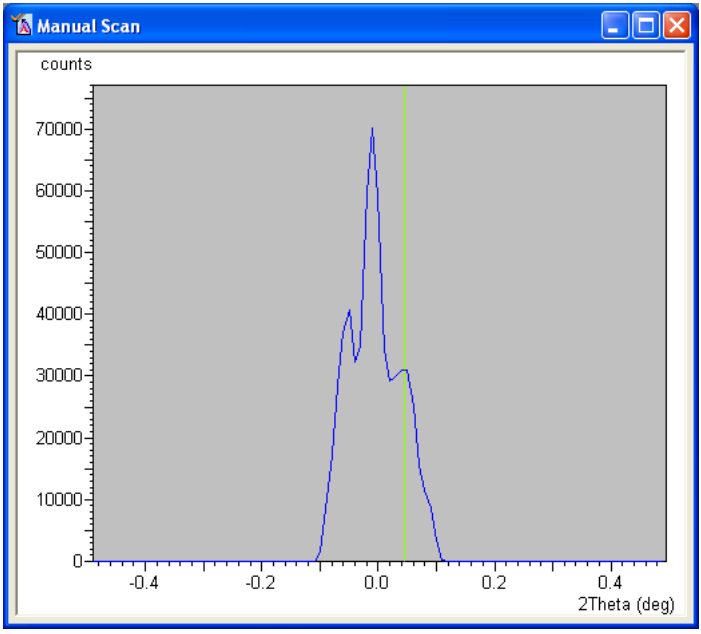
We will now make a short manual scan to ensure that the beam is located exactly at 0° 2Theta (= maximum count rate).

- In the instrument control window "Instrument Settings" tab, check that the positions for: "2Theta", "Omega", "Phi" and "Psi" are all zero and press OK.
- Select *Measure – Manual Scan*.
- Select "2Theta" from the "Scan axis" drop-down list. In the "Scan mode" frame select the "Continuous" radio button and the following scan parameters:

"Range (°):"	0.9900
"Step size (°):"	0.0100
"Time per step (s):"	0.50
"Scan speed (°/s):"	0.020000



- Press Start. After a while a manual scan result window will be displayed.



- If the peak is not central as in this example: grab the hairline, move it to the peak (or as near to it as you can) and press Start.

- Move the hairline as follows:
  - Click the right mouse button.
  - Select *Move mode*.
  - Grab the hairline and move it to the center of the peak.
- If necessary continue moving the hairline and pressing Start until you have moved the hairline to the center of the peak.
- Make a note of the count rate at the peak. You can see that by moving the + cursor to the top of the peak and reading the count rate in the status bar (approximately 770 in this example).
- Press  on the Manual Scan and Prepare Manual Scan window.
- Double-click on Beam Attenuator in the "Incident Beam Optics" tab.

  Set "Usage:" to "Do not switch" and check (✓) "Activated".
- Press OK.
- Open the shutter by pressing the  button on the Toolbar.
- Double-click on the "Positions" branch in the "Instrument Settings" tab on the instrument control window.
- Now you must change the Z position until the displayed count rate is about half the total you noted when you were moving the hairline in the previous step.

  In this example we have a count rate 770 (rounded up) and we need to move the sample until the measured count rate = half that value (385). This is an iterative process as with the following example:

  Enter 7.5 into the "Z" Box, press Apply result = 770

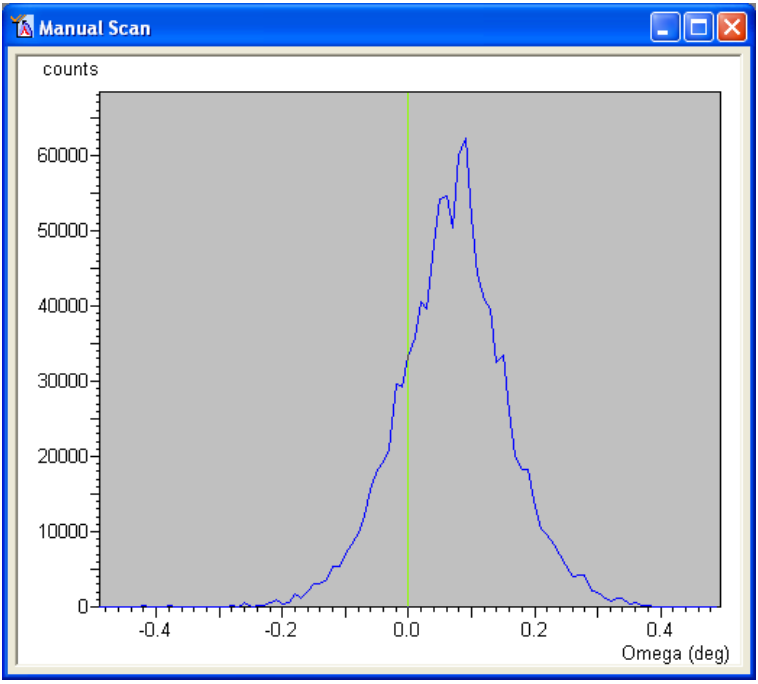
  Enter 8 into the "Z" Box, press Apply result = 0

  Enter 7.75 into the "Z" Box, press Apply result = 730

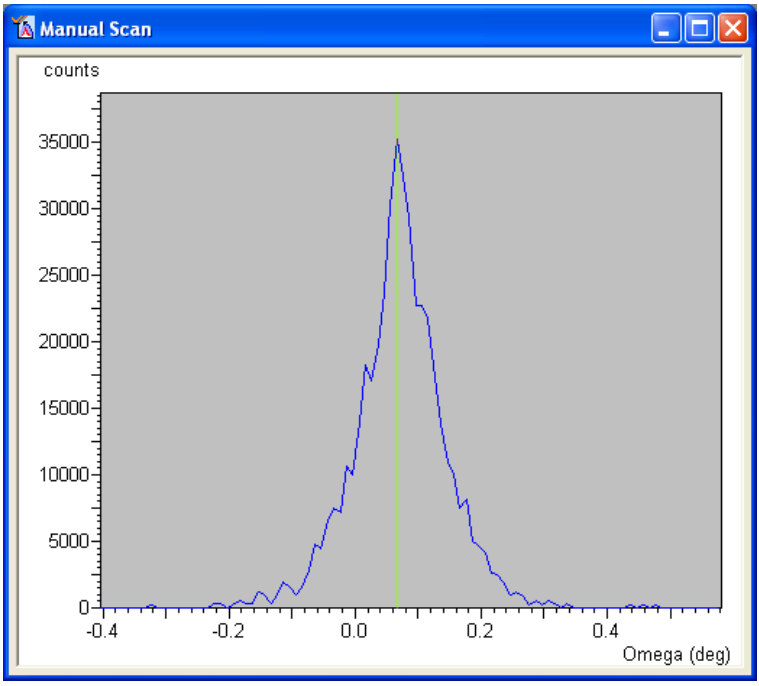
  Enter 7.907 into the "Z" Box, press Apply result = 10

  Enter 7.798 into the "Z" Box, press Apply result = 385
- When you are satisfied that you are at half the count rate press OK.

- Select *Measure – Manual Scan* and then on the "Prepare Manual Scan" window select "Omega" and press Start.
  After a short while a "Manual scan" result window will be displayed.



- Change to Move mode and move the hairline to the position with highest intensity and wait for a few moments until the goniometer has moved to that position and then minimize the "Manual Scan" and "Prepare Manual Scan" windows.
- In the instrument control window double-click on the "Positions" branch and once again change the Z position until the count rate nearly equals half the first noted value (in this example: 385) and press OK.
- Restore the "Manual Scan" window and re-do the scan by pressing the right mouse button and then Start.
- If necessary, move the hairline to the center of the peak again.



There are three criteria that must be met at this stage:

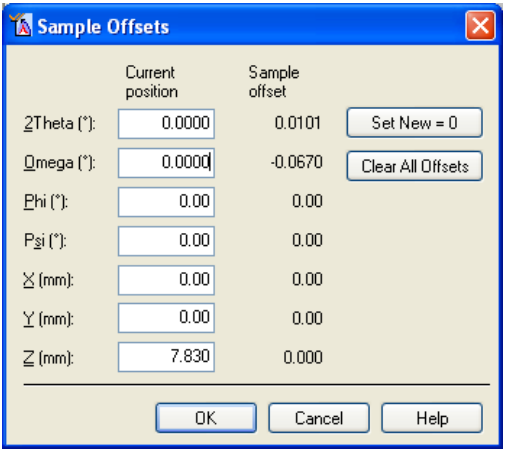
- The peak should be triangular (the scan above is a good example). You can zoom in on the peak for a closer inspection to make sure that it really is triangular.
- The hairline must be in the center of that peak.
- The count rate at the center of the peak (where the hairline is) should be half the original value that you noted.

• If your peak does not meet all three criteria, continue with the iterative procedure until it does.

4.6.5 Aligning the Sample

At this time you need to tell the goniometer exactly where the surface of the sample is parallel to the incident beam ("zero" the goniometer).

- Select *User Settings – Sample Offsets…*
- Enter 0.0000 in the "Current position" fields for "2Theta" and "Omega" (this creates a Sample offset).

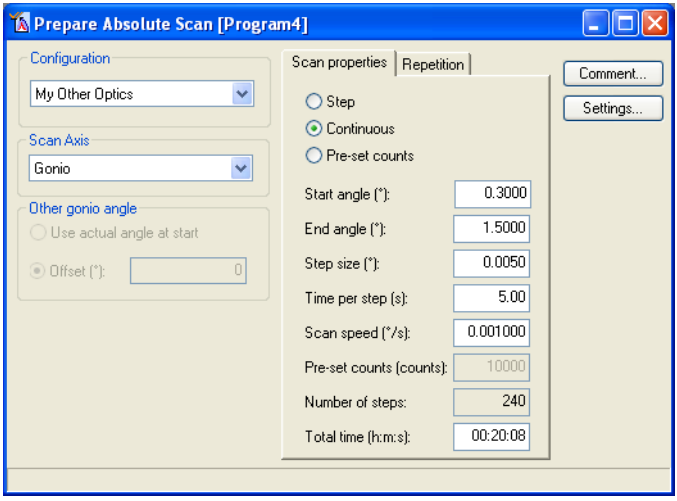


- Press OK.
- Press on both of the Manual Scan windows.

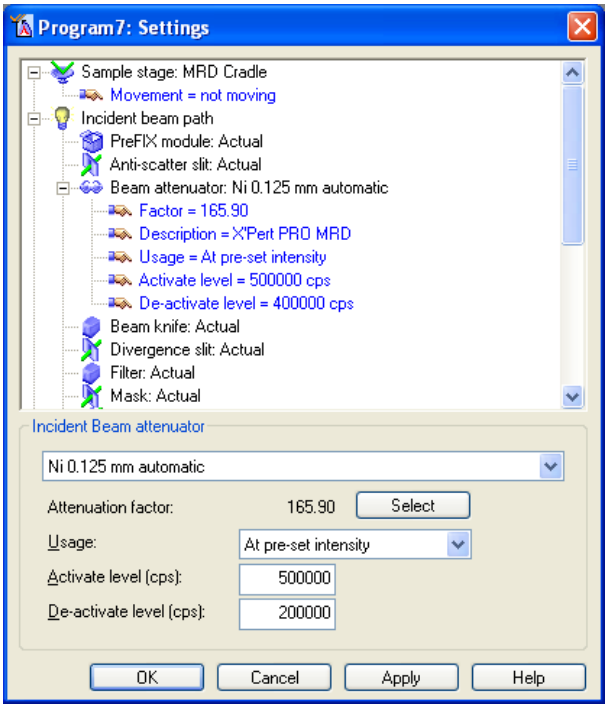
4.6.6 Create Measurement Program

- Select *File – New Program…*
- Choose "Absolute scan" and press OK.
- Enter the following parameters:

  Scan axis: Gonio
  Start angle (°): 0.3000
  End angle (°): 1.5
  Step size (°): 0.005
  Time per step (s): 5



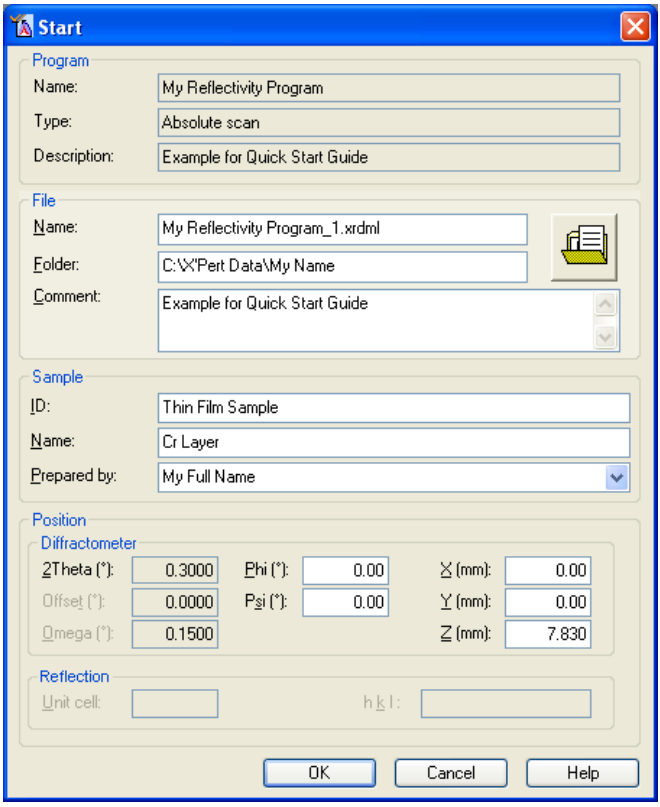
- Press Settings...
- On the "Incident beam path" branch click on beam attenuator and choose the beam attenuator that your system has in the beam path from the drop-down list (in this example: "Attenuator Ni 0.125 mm automatic").
- Set the Usage to "At pre-set intensity", the Activate level to 500000 and the De-activate level to 200000.
- Make sure that the right attenuation factor is selected.



- Press Apply and then OK.
- Select *File – Save As…*
- Enter a name for the program (in this example: "My Reflectivity Program") press OK and then close the Prepare Absolute Scan window with .

4.6.7 Measuring the Reflectivity Curve

- Select *Measure – Program* and press OK.
- Enter a name, folder and comment for the XRDML file (in this example: we accept the default").
- Enter the sample ID as "Thin Film Sample", the sample name as "Cr layer" and select "My Full Name" from the "Prepared by" drop-down list.

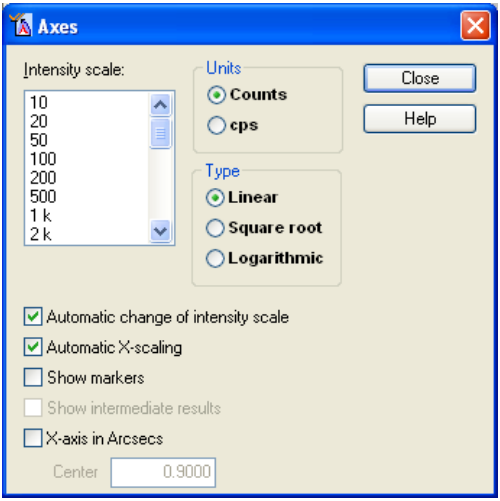


- Press OK.

4.6.8 Changing the Axes

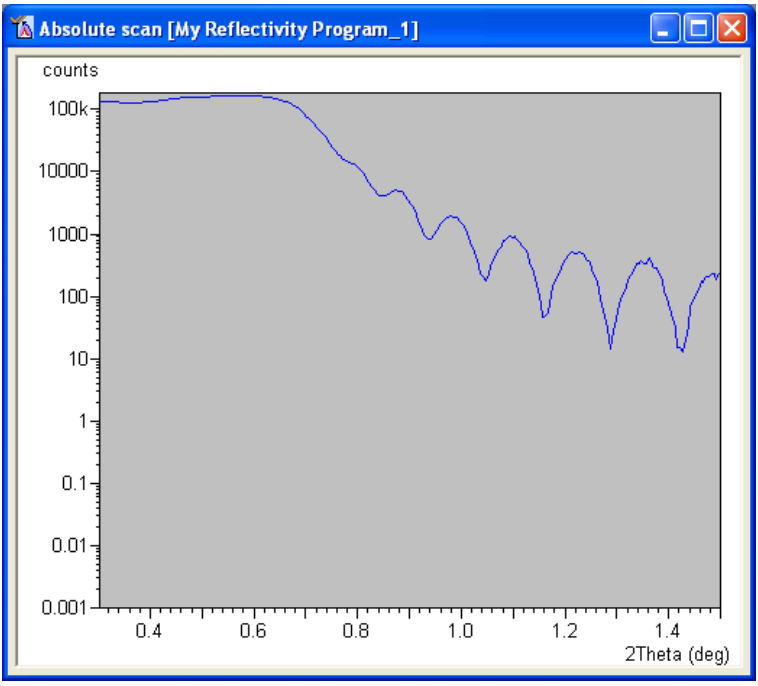
Once the scan has started you should change the scale to "logarithmic" to display the reflectivity curve better.

- Click the right mouse button in the graph window and choose *Axes*….



- Select: Units: Counts
  Type: Logarithmic
  Check "Automatic change of intensity scale"
  Check "Automatic X-scaling"
- Press Close.

The resulting curve can be used to calculate the thickness of the film measured.



You have now collected the data.

You can now use X'Pert Data Viewer to view your results. A guide to using X'Pert Data Viewer is given in the X'Pert Explorer Add-ons Quick Start Guide (4022 339 07591).

If you want to automatically use the results of future measurements, you could for example, utilize one of the scripts available with X'Pert Automatic Processing Program. Examples of these scripts and of how to use them is given in the X'Pert Automatic Processing Program - Quick Start Guide (4022 339 07891).

- Go off-line by selecting *Instrument* - *Disconnect* and then press OK.

X'Pert Data Collector - Quick Start Guide

PANalytical

Chapter 5

Using X'Pert Data Collector with X'Pert PRO MPD

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X'Pert Data Collector - Quick Start Guide

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

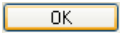
The system that we will use to gather the data is an X'Pert PRO MPD crystallography configuration for phase analysis comprising:

Sample Stages:	Sample spinner
	Capillary spinner
Incident Beam Optics:	Curved monochromator
	X-ray mirror
	Hybrid monochromator
	Programmable optics
Diffracted Beam Optics:	Programmable optics
	Parallel beam collimators
	X'Celerator detector.

5.2 SYSTEM PREPARATION

5.2.1 Starting the Software

- Double-click on the X'Pert Data Collector icon 

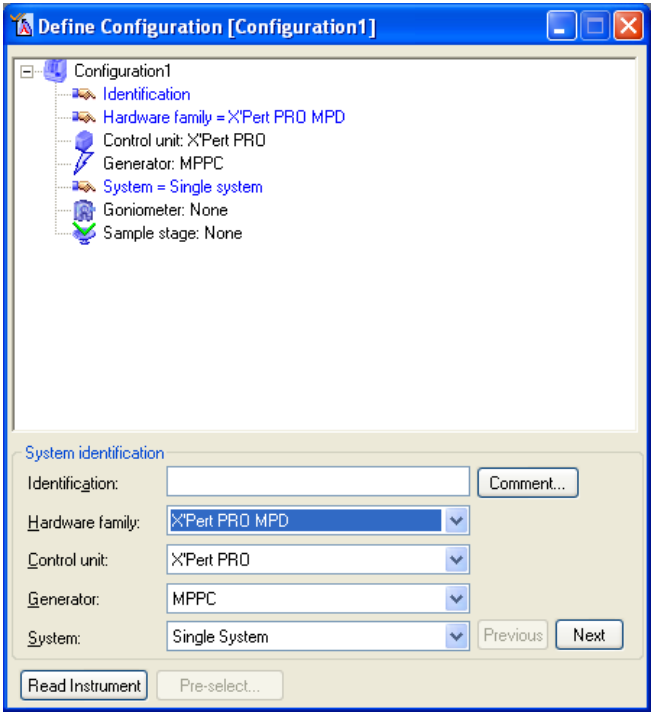
.
- Enter the user name and password: "My Name" and "password" and press 

.

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).

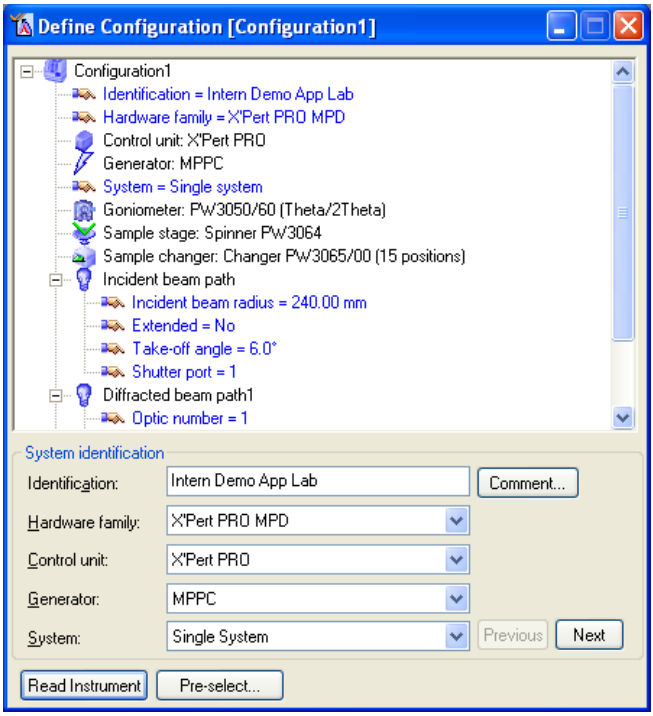


- Find out what hardware is known to the instrument control software by pressing Read Instrument.

  If the system has not been previously initialized an initialization wizard will start. This wizard is designed to handle situations where there is a possibility of collision when the diffractometer resets (for example: system parts might collide). If this wizard does start, just follow the instructions displayed on the screen.

- Wait until the system is ready (the "Connecting to instrument" pop-up disappears).

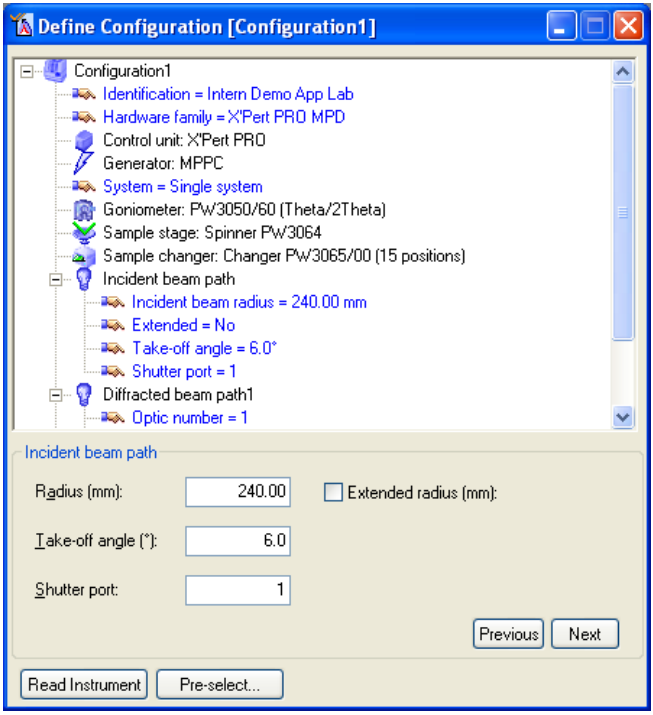
  If the system cannot uniquely identify some of the items it will ask you to specify what you have.



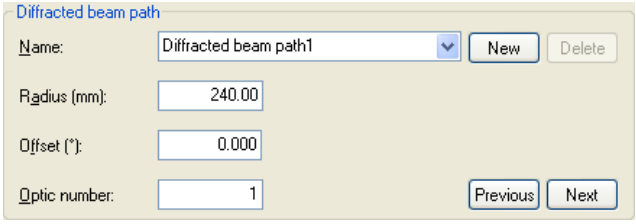
- Press the Next button and check the items in the "Goniometer/ Sample Stage" frame.

  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. If you have a non-ambient sample stage you have to select the controller using the Controller... button.

- Press the Next button to see the "Incident beam path" description.

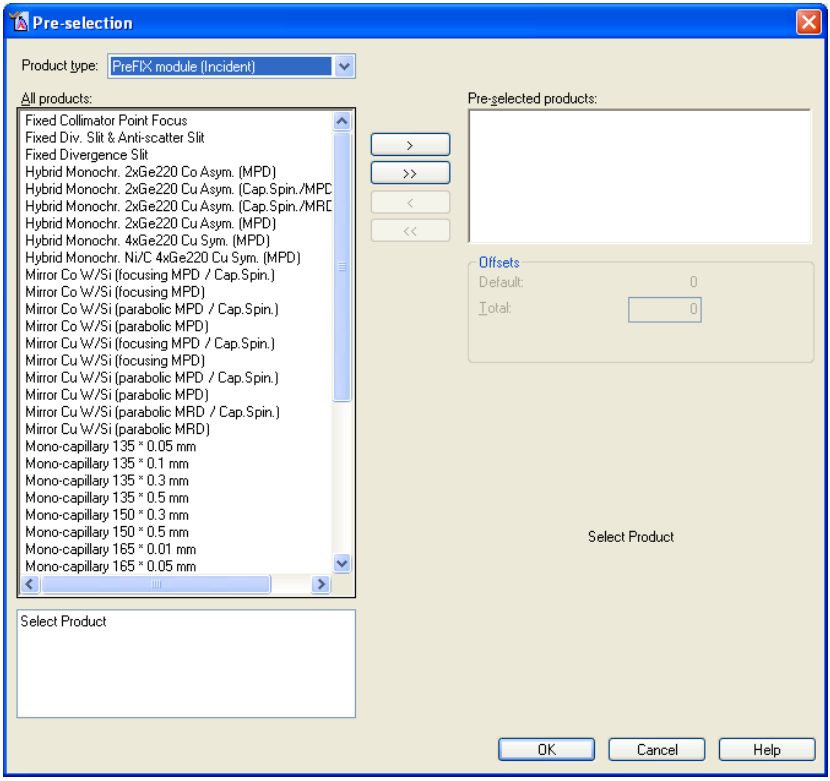


- Confirm that the information displayed here is correct.
- Press the Next button to see the diffracted beam path description(s).



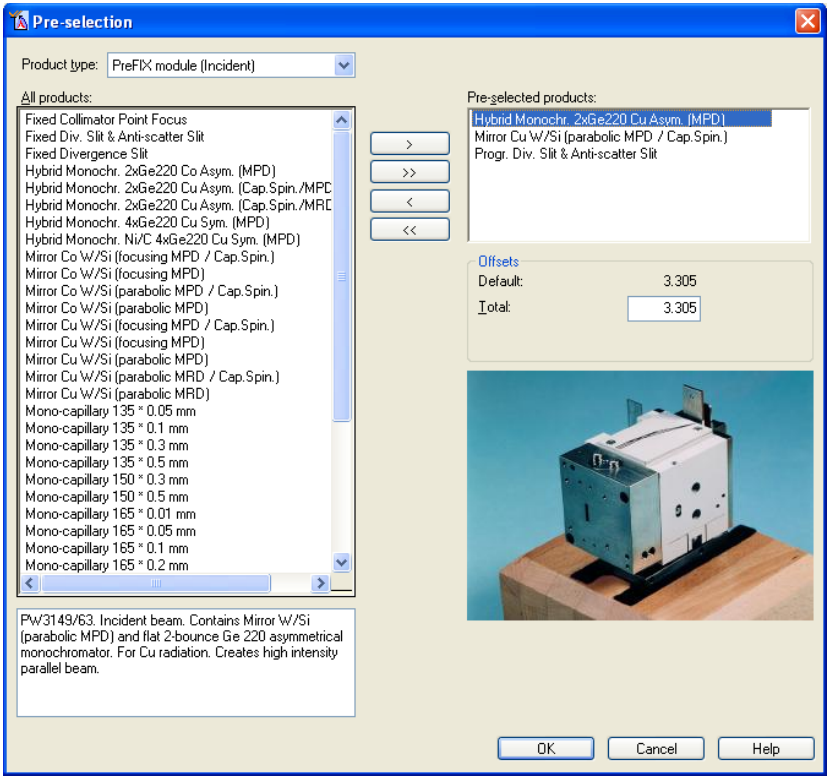
- Confirm that the information displayed here is correct (don't forget to check all diffracted beam paths using the drop-down list).

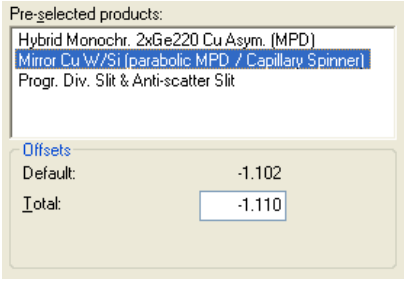
- Press the Next button to see the "Axes" frame for defining the limits and manual load positions which we will not change at this time.
- Press the button Pre-select... to obtain a series of pages where you can tell the system what changeable devices are available to you. Always start with the PreFIX module (Incident), the default, and then the PreFIX module (Diffracted) products if any are available.



To select an item, you highlight that item in the "All products" list and then press > .

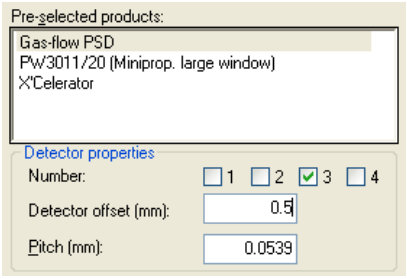
If you have X-ray Mirror or Hybrid monochromator PreFIX modules in your system it is important that you enter their offsets that are provided in the System Acceptance Form delivered with your system.



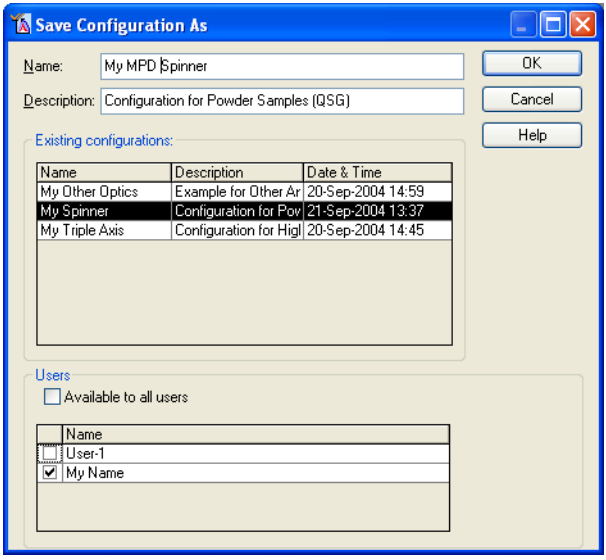


- Leaf through each entry in the "Product type" drop-down list and select those products that are available to you.

If your system includes an X'Celerator detector (and an X'Celerator detector monochromator) or a position sensitive detector it is important to define the "Detector offset" provided in the System Acceptance Form delivered with your system.

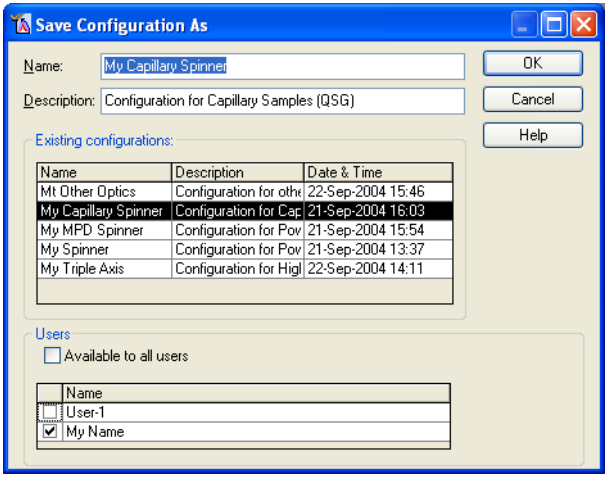


- When you have selected all of the items that you require press OK.
- Save the configuration by selecting *File - Save As....* In this example we saved the configuration as "My MPD Spinner".



- Press OK.

- If you have more than one sample stage (for example a spinner and capillary spinner), 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 Previous button three times.
- In the "Goniometer/Sample stages" frame select the next sample stage from the drop-down list.
- Select *Tools - Exchange Sample Stage* and proceed as described in section 5.4.1 to actually change to the required sample stage.
- Press Read Instrument 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 Capillary Spinner". This saves the same configuration again with the exception of the new sample stage.



- Press OK.
- Repeat these actions for the remaining sample stages and save the configurations.
- Exit this phase by pressing .

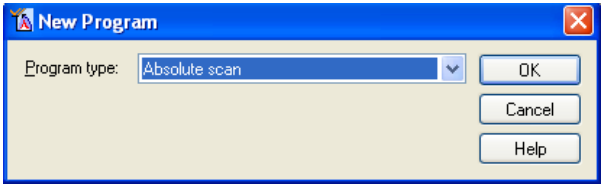
5.3 SILICON SAMPLE

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/X'Celerator PreFIX module with PASS on the diffracted beam side.
- Switch the system on by pressing the "Power On" button on the enclosure's console. When the kV display shows 30 and the mA display 10, the system is ready for use. If the power does not run to 30 kV and 10 mA please refer to your X'Pert PRO System User's Guide.

5.3.2 Creating the Measurement Program

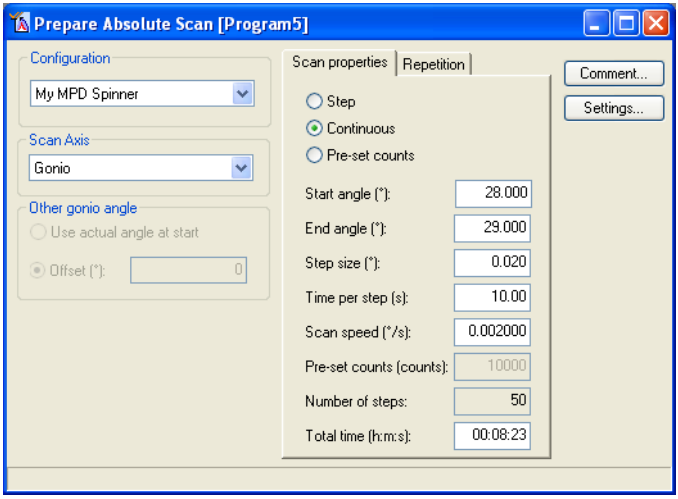
- Select *File - New Program.*



- Select "Absolute scan" from the "Program type" drop-down list and press OK.
- Make the following entries:

Configuration	"My MPD Spinner" (from the drop-down list). You may get a message telling you that "The settings will be cleared for this configuration", in this case press OK.
Diffracted beam path	If applicable (if you have more than one Diffracted beam path), choose the diffracted beam path containing the X'Celerator PreFIX module.

Scan Axis	Gonio
Start angle	28
End angle	29
Step size	0.02
Time per step	10.00



- 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 module: Actual" and select the "Prog. Div. Slit Module & Anti-scatter Slit" from the drop-down list and press Apply.
- 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 "Mask Fixed 20 mm (MPD/MRD)" from the drop-down list.
- Click on "Monochromator" and select "Inc. Beam 1xGe111 Cu/Co (a1 for reflection mode)" from the drop-down list.

In the Diffracted beam path:

- Click on "PreFIX module:" and change it to "X'Celerator".
- Click on "Detector" and select "X'Celerator".

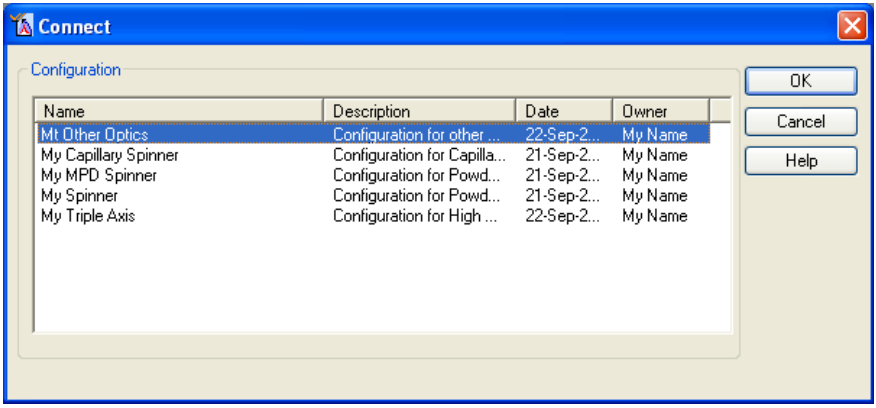
  Set the "Scan Mode" to "Scanning".

  Set the "Active length (º2Theta):" to "0.5" (the system will set it to the nearest allowed value).
- Click on the "Anti-scatter slit", select the "Prog. AS Slit" and set it to: 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 .

5.3.3 Performing the Measurement

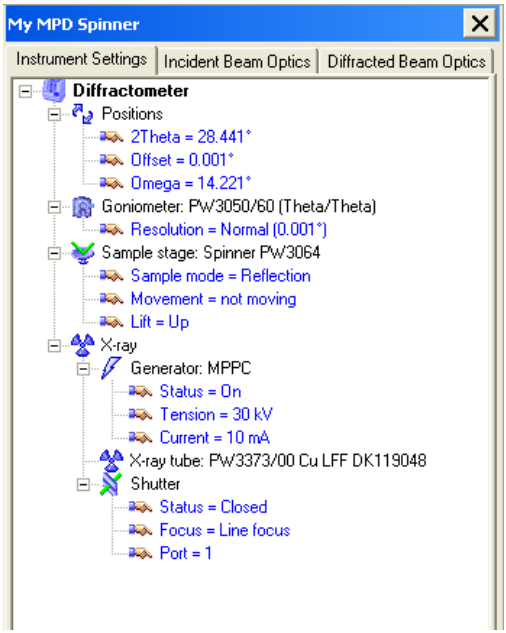
If the correct sample stage is not mounted, use *Tools - Exchange Sample Stage* as described in section 5.4.1 to change to the required sample stage.

- Select *Instrument - Connect*.
- Choose the correct configuration and, if applicable, the required beam path.

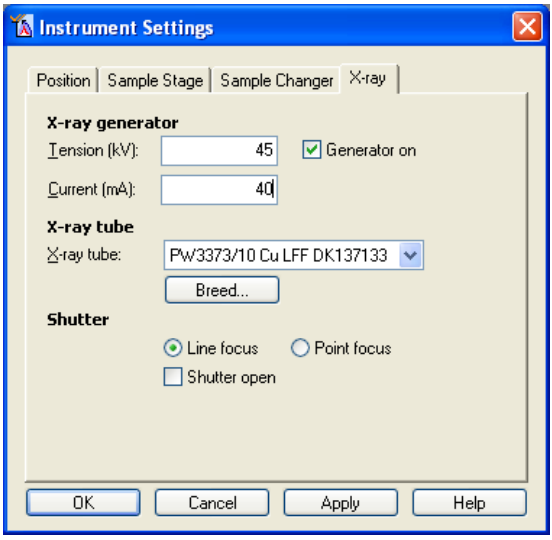


- Press OK.

If you get any system instructions follow them.



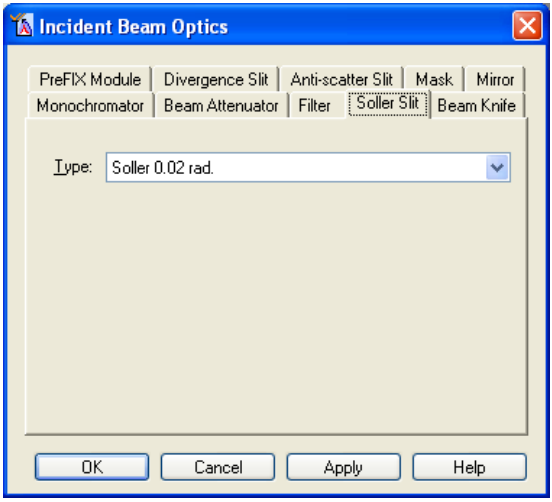
- Double-click on "Generator", set the power to "45 kV" and "40 mA".



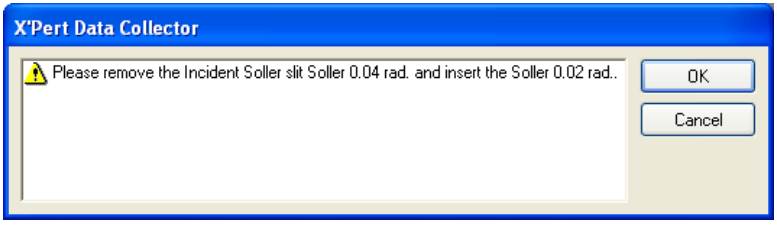
- Press Apply.
- Depending on your situation, mount the Si sample as follows:

If you do not have a sample spinner in your system:	Mount the sample on the sample stage, close the enclosure doors and press OK.
If you do not have a sample changer in your system:	Select the "Sample Stage" tab. Use the handle to lower the sample spinner platform, mount the sample, release the handle to bring the sample to the spinning position. Close the enclosure doors and press OK.
If you have a sample changer, but it is positioned in the corner:	Select the "Sample Stage" tab. Uncheck "Lift Up", close the doors and press Apply. Open the doors, mount the sample, close the enclosure doors, check "Lift Up" and press OK.
If you have a sample changer, and it is ready to use:	Select the "Sample Changer" tab. Open the doors, put the sample into an empty magazine or monitor position. Close the doors, indicate to load the sample from the position you just loaded the sample into and press OK.

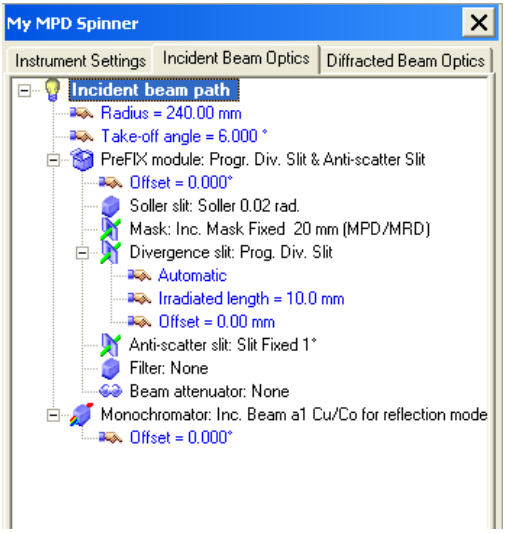
- Select the "Incident Beam Optics" tab.
- Double-click on the "Incident beam path" and then select the items in the beam path by selecting the relevant tabs and the types from the drop-down lists, starting with the PreFIX module and pressing Apply before doing the rest of the changes.



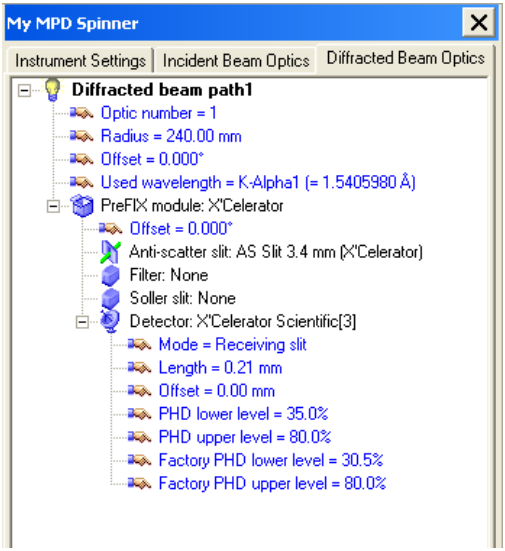
- Press OK.
- If there are any system instructions, follow them.



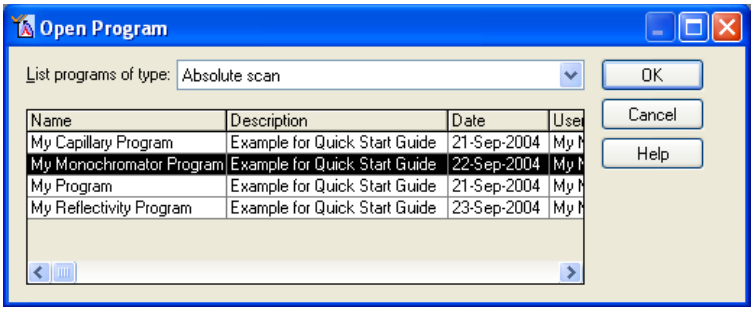
- Press OK.



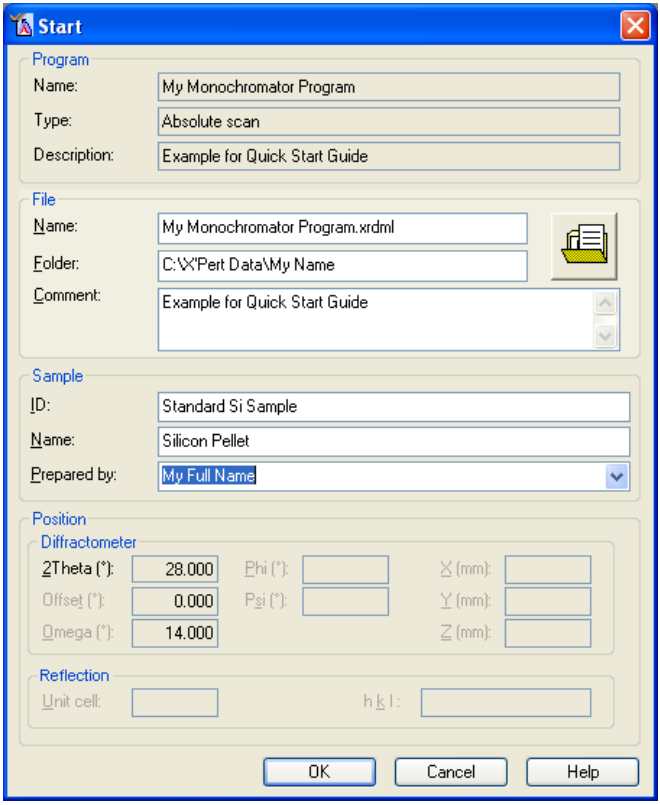
- Select the "Diffracted Beam Optics" tab.
- Double-click on the previously selected diffracted beam path ("Diffracted Beam Path1") and then select the items in the beam path by selecting the relevant tabs and the types from the drop-down lists, starting with the PreFIX module and pressing Apply before doing the rest of the changes.
- Press OK.
- If there are any system instructions, follow them.



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

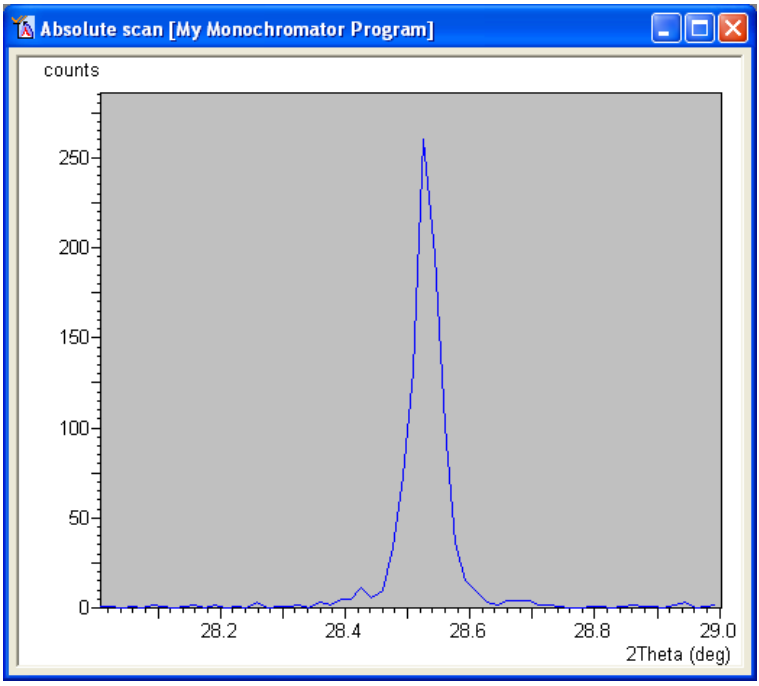


- Press OK.
- Enter a file name, the folder where it should be located, and optionally, a comment. Enter: sample ID: "Standard Si Sample", sample name: "Silicon Pellet", and select "My Full Name" from the "Prepared by:" drop-down list.



- Press OK.

  The scan results are displayed as the measurement progresses.



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You can now use X'Pert Data Viewer to view your results. A guide to using X'Pert Data Viewer is given in the X'Pert Explorer Add-ons Quick Start Guide (4022 339 07591).

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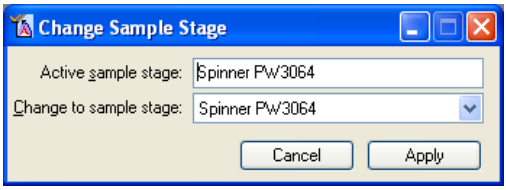
- Go off-line by selecting *Instrument* - *Disconnect* and then pressing OK.

5.4 CAPILLARY SAMPLE

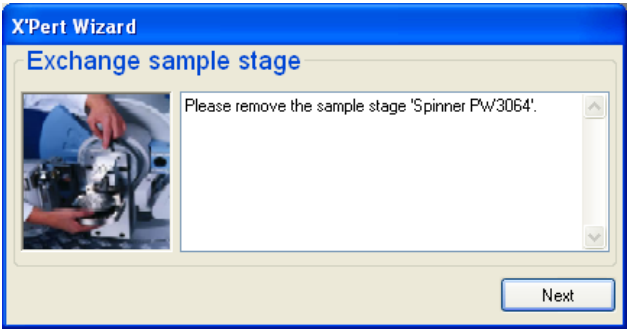
We are going to measure a capillary sample filled with an unknown powder. A "standard" capillary sample is not delivered with the system, therefore you will have to provide your own sample to do this example. This means that the data that you collect will of course be different to that given here.

5.4.1 Preparation

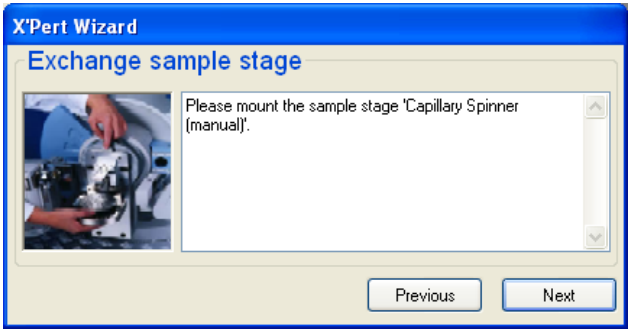
- Mount the capillary spinner:
- Select *Tools - Exchange Sample Stage* and wait for the system to connect to show the first screen in this wizard. In the previous example we had a spinner fitted (see section 5.3) and we need to change it to a capillary spinner.



- Select Capillary Spinner (Manual) from the drop-down list and press Apply.
- If everything is correct press Next.



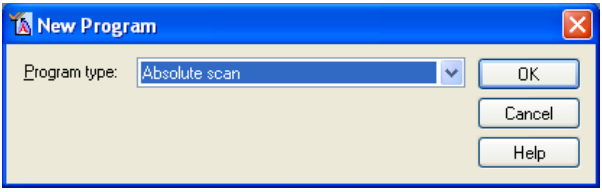
- Remove the spinner and press Next.



- Mount the capillary spinner and press Next. The system will acknowledge that it is ready for use. Press Finish.
- If you have a hybrid monochromator or an X-ray mirror (in this example: a hybrid monochromator), mount them.
- Mount the capillary sample.

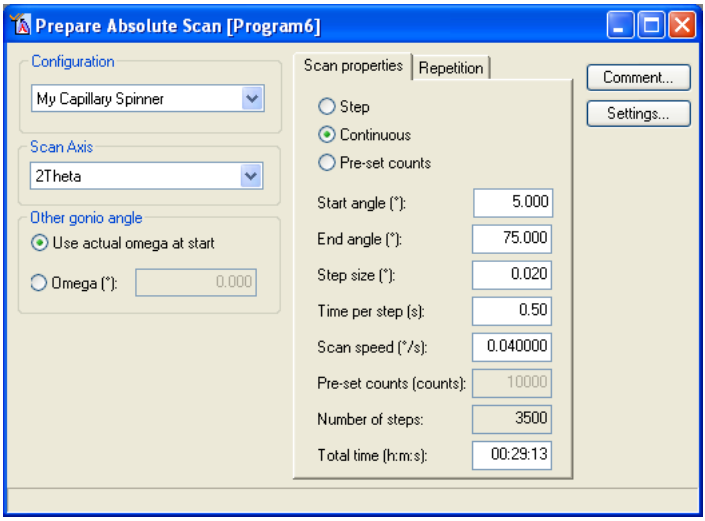
5.4.2 Creating the Measurement Program

- Select *File - New Program*

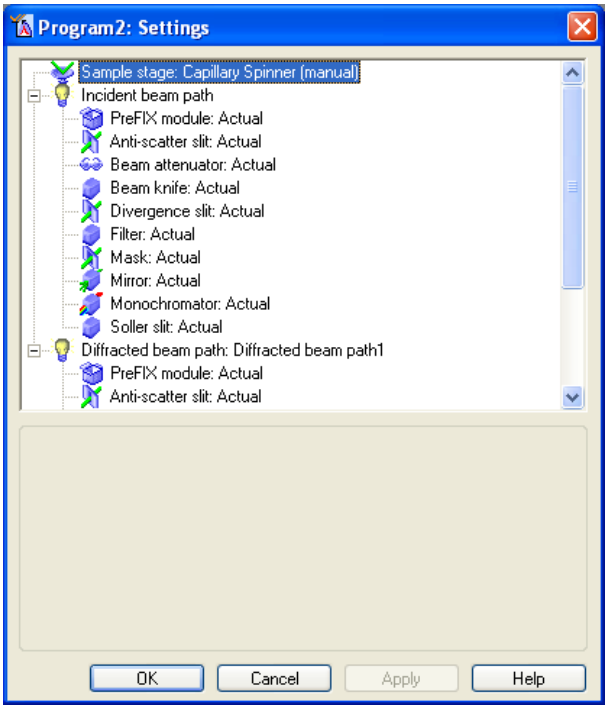


- Select "Absolute scan" from the Program type drop-down list and press OK.
- Select "My Capillary Spinner" from the Configuration drop-down list.
- If applicable, make sure that the correct diffracted beam path is selected.
- You may get a message "The settings will be cleared for the diffracted beam path <name of your beam path>". In this case press OK.

- Select the required Scan Axis ("2Theta" in this example).
- Select the required Scan mode ("Continuous" in this example).

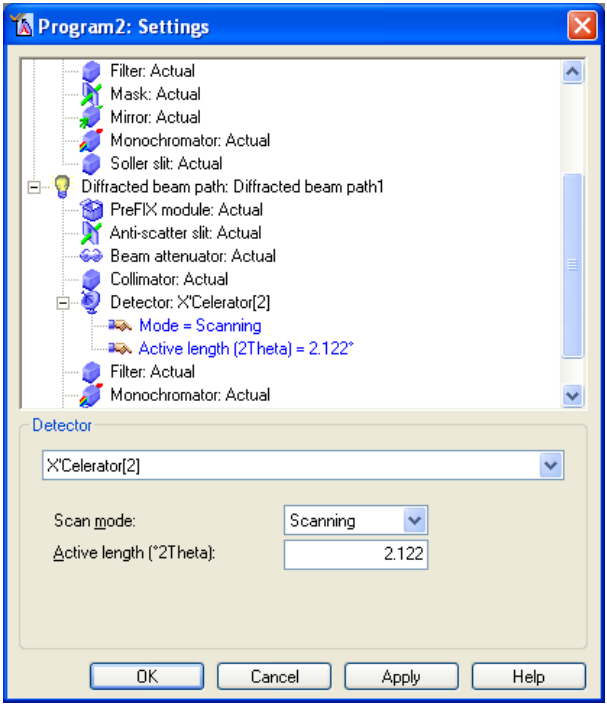


- Press Settings....

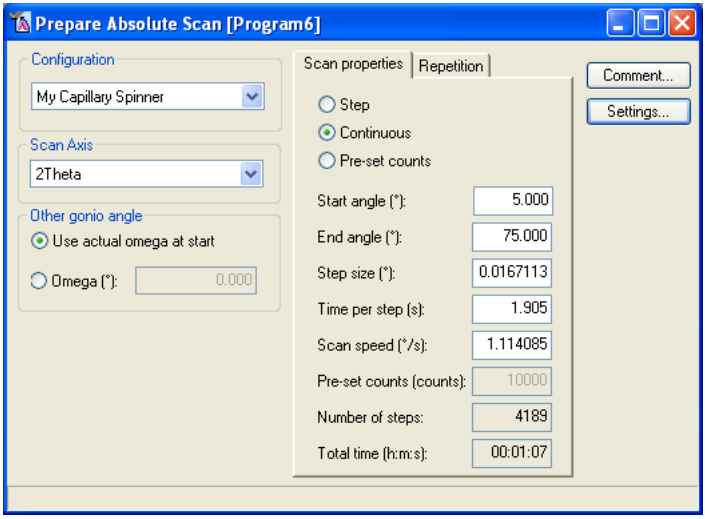


- Click on the "Detector" icon (if necessary slide down the scroll bar until the icon is visible).
- Change the settings from actual to X'Celerator.

Select:	Scan Mode:	Scanning
	Active length (º2Theta):	2.122 (or the maximum allowed in your system)


Program2: Settings
Filter: Actual
Mask: Actual
Mirror: Actual
Monochromator: Actual
Soller slit: Actual
Diffracted beam path: Diffracted beam path1
PreFIX module: Actual
Anti-scatter slit: Actual
Beam attenuator: Actual
Collimator: Actual
Detector: X'Celerator[2]
Mode = Scanning
Active length (2Theta) = 2.122°
Filter: Actual
Monochromator: Actual
Detector
X'Celerator[2]
Scan mode:
Scanning
Active length (°2Theta):
2.122
OK
Cancel
Apply
Help

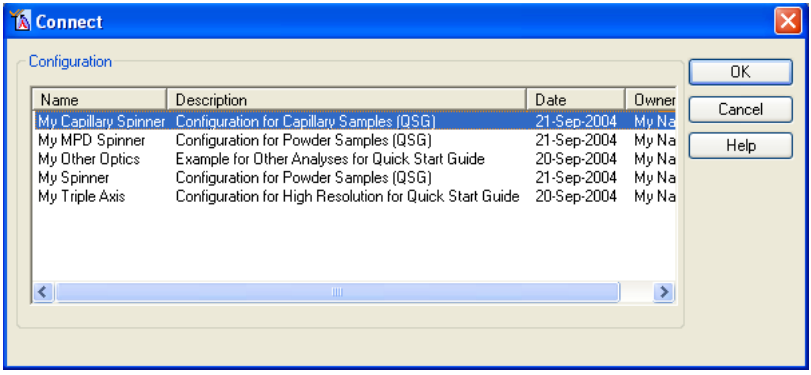
- Press OK.



- The program is now ready, save it as "My Capillary Program" and press  to finish this step in the procedure.

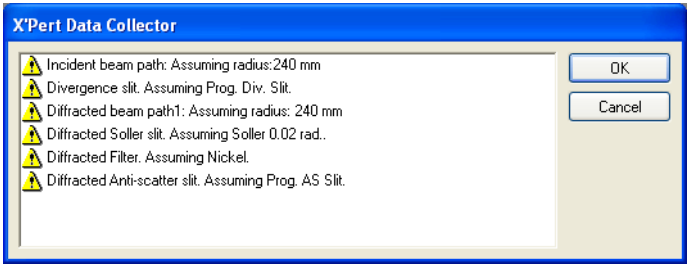
5.4.3 Performing the Measurement

- Go on-line by selecting *Instrument* - *Connect.*

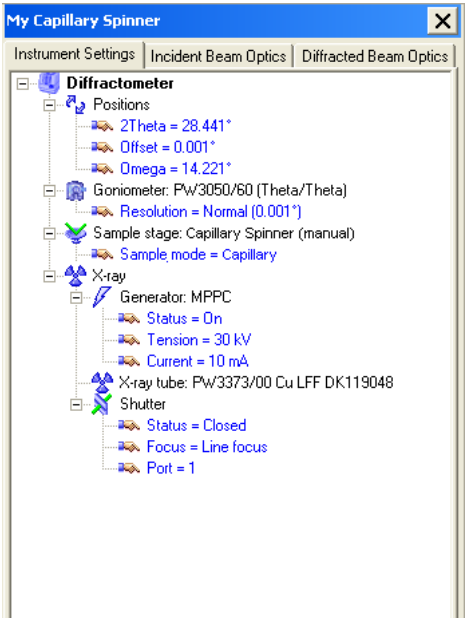


- Choose the configuration you want to use (in this example: "My Capillary Spinner") and, if applicable, the beam path containing the X'Celerator.

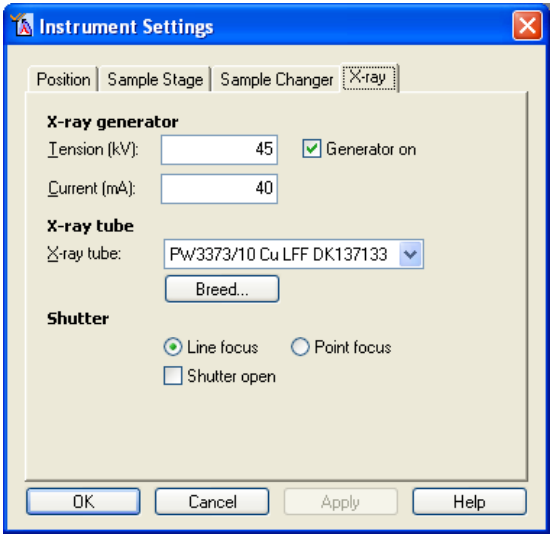
  A message window showing the 'assumed' status of the system is displayed:



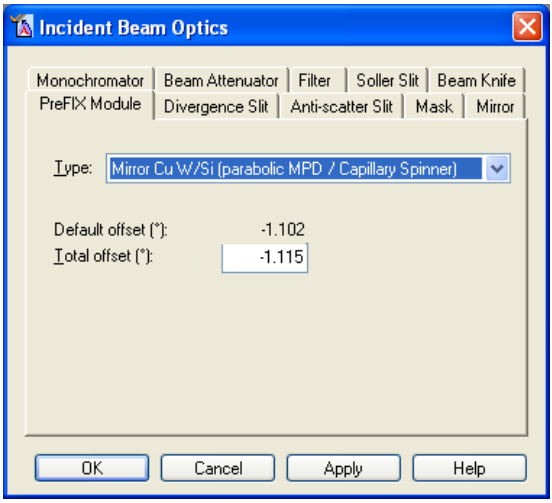
- In order to make sure that you obtain a good measurement, you must carefully check these assumptions. If these assumptions are correct, press OK and proceed with the next step. If they are not correct you must still press OK and then go to the tab(s) on the instrument control window containing the incorrect assumption and make the corrections.
- Carefully look through the "Instrument Settings", "Incident Beam Optics" and "Diffracted Beam Optics" tabs to make sure that you have the correct parts mounted.



- Set the Power:
- Double-click on the "Generator" icon and enter the required power settings: "45 kV" & "40 mA".

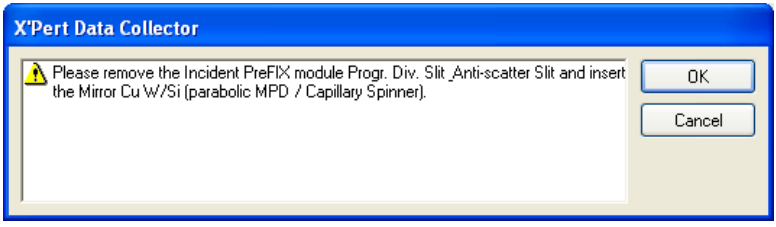


- Press OK.
- Select the "Incident Beam Optics" tab.
- Double-click on the "Incident beam path" and then select the PreFIX module that you want to use, press Apply.
- If there are any, perform the actions as instructed and press OK.
- Select the items in the beam path by selecting the relevant tabs and the types from the drop-down lists.

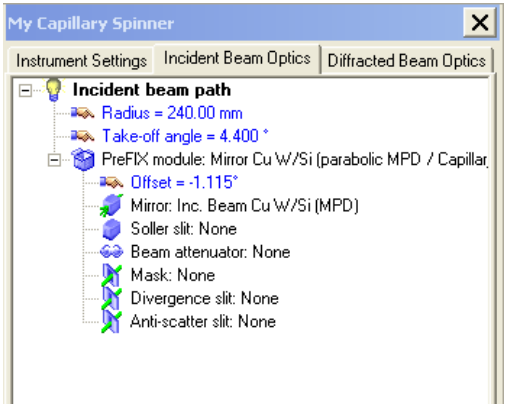


- Eventually, when you have selected all the items in the incident beam path, press OK.

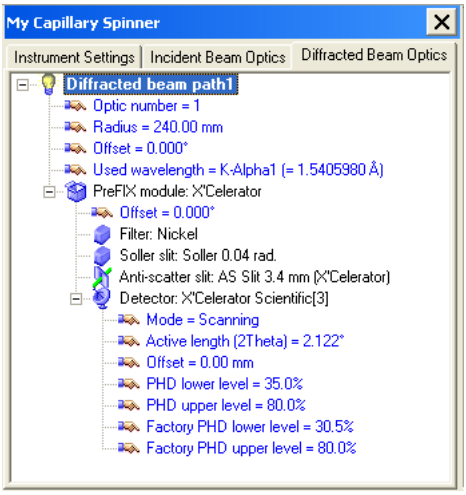
  You may get some instructions to follow, for example: "Please remove the Incident PreFIX module Progr. Div. Slit Anti-scatter Slit and insert the Mirror Cu W/Si (parabolic MPD / Capillary Spinner)".



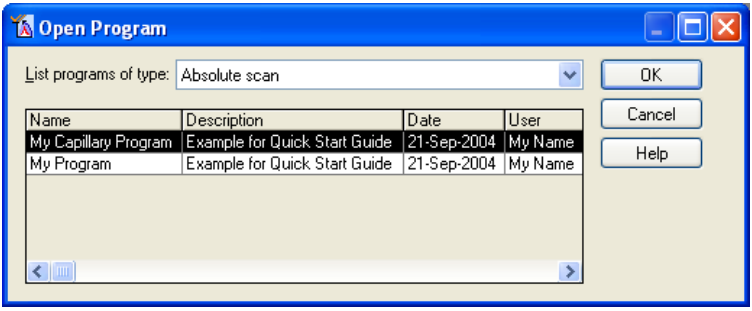
- Either perform the actions as requested and tell the system when they are done by pressing OK, or if the actions had already been done, just press OK.



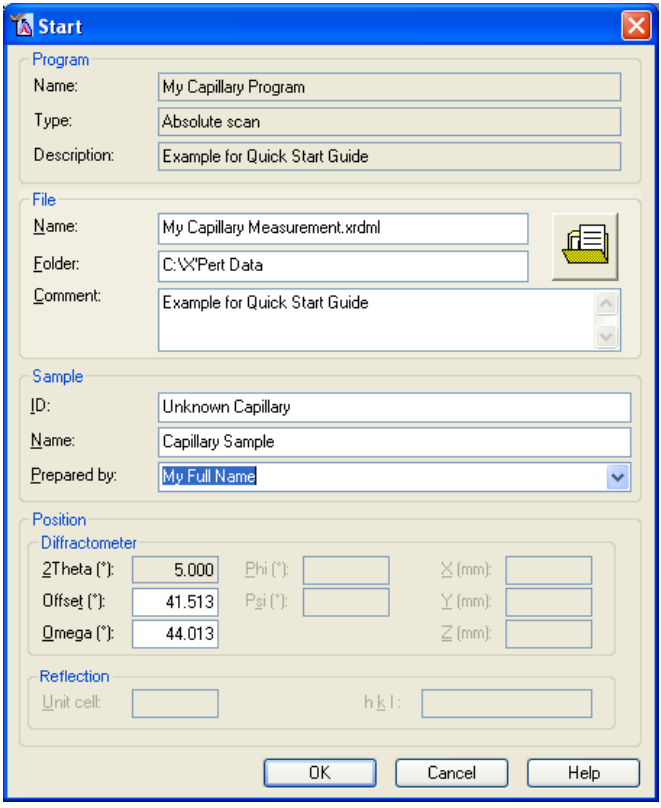
- Select the "Diffracted Beam Optics" tab.
- Double-click on the relevant beam path, in this example: "Diffracted beam path1" and then select the items in the beam path by selecting the relevant tabs and the types from the drop-down lists, starting with the PreFIX module and pressing Apply before doing the rest of the changes.
- Eventually, when you have selected all the items in the diffracted beam path, press OK. You may be asked to make some changes, if so, make the changes and confirm with OK.



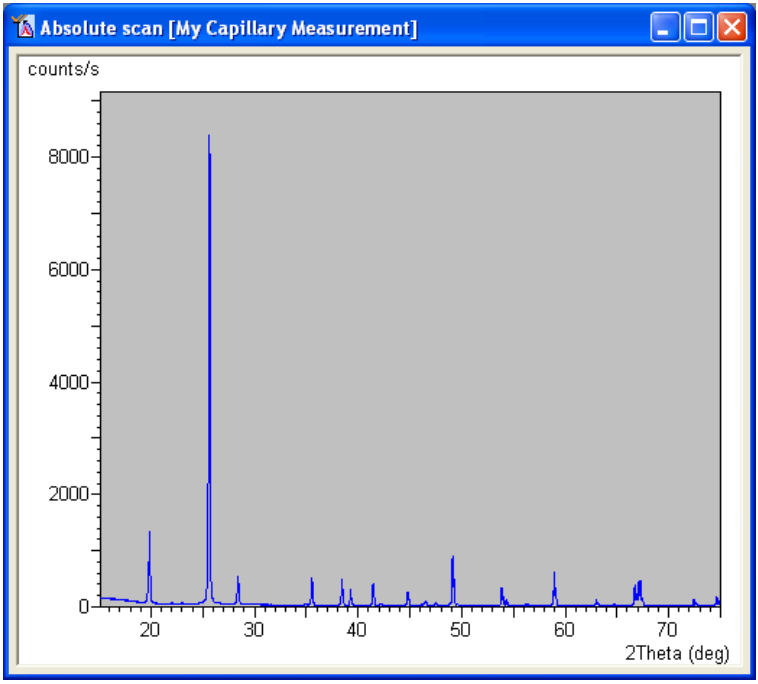
- Choose *Measure - Program*.
- Select "My Capillary Program".



- Press OK.
- In the "Start" window, enter a file name (in this example: "My Capillary Measurement.xrdml"), the folder where it should be located, and optionally, a comment. A sample ID (in this example: "Unknown Capillary"), a sample name ("Capillary Sample"), and select "My Full Name" from the "Prepared by:" drop-down list.



- Make sure that the capillary spinner is switched On and that the enclosure doors are closed and then press OK.
  The scan results are displayed as the measurement progresses.



You have now collected the data.

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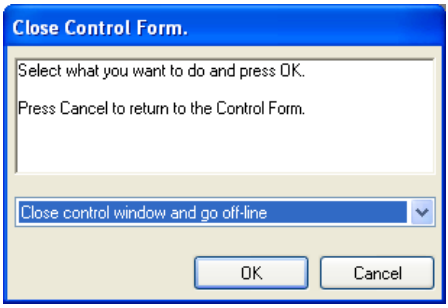
- When the measurement is completed press to clear the screen.
- Choose *Instrument* - *Disconnect* to go off-line.



- Press OK.
- Press to leave X'Pert Data Collector.

X'Pert Data Collector - Quick Start Guide

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