

X'PERT DATA COLLECTOR





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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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Chapter 1. Introduction

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.



Chapter 1. Introduction

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).			
ОК	In the examples in this Guide we terminate most actions by saying "press OK", you can if you prefer press <i>Enter</i> instead of OK.			
×	The instruction to click (or press) \boxtimes 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

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.



Chapter 2

Starting to Work with X'Pert Data Collector

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2.2	Starti	ing X'Pert Data Collector	
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	2.2.2	Normal Use	
	2.2.3	Organizing your Result Data	
	2.2.4	Report Folder	



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
 Collector

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

(į)	You cannot use this application with the current version of your database. The application expects version 7 build 2 date 02-Feb-2006. The database reports version 6 build 1 date 16-Nov-2004. Please convert your old database with the conversion program.
	ОК

- 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
- Press the <u>Yes</u> button to make a backup:

Either accept the proposed name or rename the database and press

Save I. If a message about the disk space appears and you have enough disk space press OK.

When the message about the version appears: ٠

Convers	ion 🛛
?	Input database C:\Program Files\PANalytical\X'Pert Data Collector\xpert32.db version 6 build 1 date 16-Nov-2004 (X'Pert Software version: 2.1x) Conversion to version 7 build 2 date 02-Feb-2006 (X'Pert Software Version: 2.2x) Conversion is about to begin. This may take some time. Do you want to continue?

press <u>Yes</u> and the following screen appears:

## Database Conversion				
Table Name	Bows	Time	Bow 🔼	Chart
	0	0	0	Juli
ANODE_MATERIAL	0	0	0	Abort
CODE_TABLE	0	0	0	
AST_UPDATE_TIMESTAMP	1	0	1	
ELEMENT	0	0	0	Close
FUNCTIONAL_GROUP	0	0	0 📄	
	0	0	0	Information
44 DIFFRACTION_USER	18	1	18 🚽	
Status: Converting database Processing: DIFFRACTION_OBJECT			17 of 326	I I
Rows/s: 457				

Close Press as soon as the button is enabled.



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

You may get a message about incorrect conversion results:

Convers	ion 🛛
į)	Database has been converted succesfully. The X'Pert 2.1x version of the software can be used. However, 10 Q-scans could not be correctly transferred to XRDML files. These scans all have the following automatically generated comment added to the XRDML file: 'Conversion note: Q-scan points not correctly written to the XRDML file.'. Contact your local PANalytical representative if you want these scans to be correctly transferred into XRDML. You can easily obtain a list of XRDML filenames involved by using the search functionality of your Windows Explorer. (Use the text search option with the text: 'Q-scan points not correctly'). OK
•	Press OK

2.2.2 Normal Use

Login	X
User:	
Password:	
OK Canc	el

• Log on with the following information:

User: User-1

Password: galaxy

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

(۵	('Pert	Data (Collector	(User-1)					[
Eile	Edit	⊻iew		Instrume	nt <u>T</u> ools	<u>U</u> ser Settings	<u>S</u> ystem Settings		<u>W</u> indow	<u>H</u> elp
			6 X	b b	⇒ P	0 🔶 🖗 🤅	》 댁 댁 떠	?		
		_								

In this example we will create a new user.

• Select System Settings – User Management.



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

• 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.

🚯 New User	
<u>N</u> ame:	My Name
<u>F</u> ull user name	My Full Name
<u>A</u> ccess level:	System Owner 💌
User manager:	
Password:	*****
<u>R</u> e-type password:	*****
ОК	Cancel Help

Press OK

Name Full User Name User Manager Access Level Close User-1 User-1 Yes System Owner New My Name My Full Name Yes System Owner New	🚯 Users				X
Modify Delete Help	Name User-1 My Name	Full User Name User-1 My Full Name	User Manager Yes Yes	Access Level System Owner System Owner	Close New Modify Delete Help

• Check that "My Name" is included in the "Name" list and press



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.

🚯 Measurement Typ	es & Data Folders	
Single scan Multiple	scan Batches	
🗹 Absolute scan		
Default folder	C:W'Pert Data	Cancel
💌 Relative scan		Help
Default folder	C:WPert Data	
🛃 Stationary measure	ement	
Default folder	C:WPert Data	
🗹 Optimize program		
Default folder	C:WPert Data	
🗹 Q scan		
Default folder	C:W'Pert Data	

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 (<u>)</u>:

٠



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

Select Default Folder For Absolute Scan 🛛 🔋 🗙
Select folder
OK Cancel New Folder
Apply to all single scans

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

Select Default Folder For Absolute Scan	
Enter the name of the new folder C:WPert Data∖	OK Cancel
My Name	

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

r Absolute Scan 🛛 [? 🔀
TUP ale IOW5 : 2-1 : Data onverted xpert32 Vame
ame
ame 📔
ame
ame 📔
ame
ame (

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



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

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.

ĺ	X'Pert Data Collector
	The default report folder C:\X'Pert Data\Report does not exist. Do you want to create it?
	Yes No

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

🚯 Options	×
Diffractometer Hard Copies Units Miscellaneous	
Report folder	
C:\X'Pert Data\My Name	
C History	
Show recently used configurations and programs	
Clear Press this button to remove information about	
recently used configurations and programs.	
Animations	
Show positioning animation	
Show sample changer animation	
✓ Show connect animation	
Stress measurements	
OK Cancel Help	
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 \mathbf{X} .



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

۲ <u>م</u>)	('Pert	Data (Collector	(My Full Na	me)					
Eile	Edit	<u>V</u> iew		Instrument	<u>T</u> ools	<u>U</u> ser Settings	System Settings	System Maintenance	<u>W</u> indow	<u>H</u> elp
			5 X	Da 🖪 🕂	⇒ P	0	の転転回	8		

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...

🚹 Define Config	uration [Configuration2]	
Configuration	2 stion e family 9 Single system	
System identification		
Identific <u>a</u> tion:		Comment
<u>H</u> ardware family:		✓
<u>C</u> ontrol unit:		✓
<u>G</u> enerator:		×
<u>S</u> ystem:	Single System	Previous Next
Read Instrument	Pre-select	

- 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



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

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.

🚹 Define Config	uration [Configuration1]		×
Configuration Control u Control u Co	1 tion = Intern Demo App Lab e family = X'Pert PR0 MPD nit: X'Pert PR0 : Single system ter: PW3050/60 (Theta/2Theta) tage: Spinner PW3064 shanger: Changer PW3065/00 (15 positions) beam path lent beam radius = 240.00 mm nded = No e-off angle = 6.0° ter port = 1 d beam path1 c number = 1		
System identification	lutan Dana das Lab		
Identific <u>a</u> tion:	Intern Demo App Lab	Comment	
<u>H</u> ardware family:	X'Pert PRO MPD		
<u>C</u> ontrol unit:	X'Pert PRO		
<u>G</u> enerator:	MPPC 💌		
<u>S</u> ystem:	Single System	Previous Next	
Read Instrument	Pre-select		

Press the Next button.

Configuration1 Identification = Intern Demo App Lab Hardware family = X'Pett PRO MPD Control unit: X'Pett PRO Generator: MPPC System = Single system Goniometer: PW3050/60 (Theta/2Theta) Sample stage: Spinner PW3064 Sample stage: Changer PW3065/00 (15 positions)
 Incident beam path Incident beam radius = 240.00 mm Extended = No Take-off angle = 6.0° Shutter port = 1 Optic number = 1
Goniometer/Sample stage
Goniometer: PW3050/60 (Theta/2Theta)
Sample stage: Spinner PW3064 ♥ Controller ☐ Get all sample stages in instrument
Sample changer: Changer PW3065/00 (15 positions) Previous Next Read Instrument Pre-select

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

Incident beam path		
R <u>a</u> dius (mm):	240.00	Extended radius (mm):
<u>T</u> ake-off angle (*):	6.0	
Shutter port:	1	
		Previous Next

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



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

Diffracted beam path					
<u>N</u> ame:	Diffracted beam path1	New Delete			
R <u>a</u> dius (mm):	240.00				
0 <u>f</u> fset (*):	0.000				
<u>O</u> ptic number:	1	Previous Next			

• 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.

Axes	
Limits	
Manual Load Positions	
In-plane diffractometer	
	Previous

• 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.

🚯 Pre-selection	
Productions D. Charles A. L. A. Lin and	
Product type: PreFIX module (Incident)	
All products:	Pre-selected products:
Fixed Collimator Point Focus	
Fixed Div. Slit & Anti-scatter Slit	
Fixed Divergence Slit	
Hybrid Monochr. 2xGe220 Co Asym. (MPD)	>>>
Hybrid Monochr. 2xGe220 Cu Asym. (Cap.Spin./MPL	
Hybrid Monochr. 2xGe220 Cu Asym. (Lap.Spin./MRL	
Hybrid Monochi, 2x0e220 Cu Asym, (MPD)	
Hubrid Monocht, Ni/C 4xGe220 Cu Sym, (MPD)	011.1
Mirror Co W/Si (focusing MPD / Cap Spin)	Urrsets
Mirror Co W/Si (focusing MPD)	Default: U
Mirror Co W/Si (parabolic MPD / Cap.Spin.)	Iotal: 0
Mirror Co W/Si (parabolic MPD)	
Mirror Cu W/Si (focusing MPD / Cap.Spin.)	
Mirror Cu W/Si (focusing MPD)	
Mirror Cu W/Si (parabolic MPD / Cap.Spin.)	
Mirror Cu W/Si (parabolic MPD)	
Mirror Cu W/Si (parabolic MRD / Cap.Spin.)	
Mirror Lu W/Si (parabolic MRD)	
Mono-capillary 135 ° 0.05 mm	
Mono-capillary 135 × 0.2 mm	
Mono-capillaru 135 × 0.5 mm	
Mono-capillary 150 * 0.3 mm	
Mono-capillary 150 * 0.5 mm	Calast Braduat
Mono-capillary 165 * 0.01 mm	Select Floudet
Mono-capillary 165 * 0.05 mm	
Select Product	

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.



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

A Pre-selection	
Image: Pre-selection Product type: PreFIX module (Incident) All products: Fixed Collimator Point Focus Fixed Div. Slit & Antiscatter Slit Fixed Divergence Slit Hybrid Monochr. 2xGe220 Co Asym. (MPD) Hybrid Monochr. 2xGe220 Cu Asym. (Cap. Spin./MPC Hybrid Monochr. 2xGe220 Cu Asym. (Cap. Spin./MPC Hybrid Monochr. 2xGe220 Cu Asym. (Cap. Spin./MPC Hybrid Monochr. 1xGe220 Cu Asym. (Cap. Spin./MPC Hybrid Monochr. 2xGe220 Cu Sym. (MPD) Hybrid Monochr. 1xGe220 Cu Asym. (Cap. Spin./MPC Hybrid Monochr. 1xGe220 Cu Asym. (MPD) Hybrid Monochr. 1xGe220 Cu Asym. (APD) Miror Co W/Si (focusing MPD) Cap. Spin.) Miror Co W/Si (focusing MPD) Cap. Spin.) Miror Cu W/Si (focusing MPD) Miror Cu W/Si (parabolic MPD) Miror Cu W/Si (parabolic MPD) Miror Cu W/Si (parabolic MPD) Miror Cu W/Si (parabolic MPD) Miror Cu W/Si (parabolic MPD) Mono-capillay 135 * 0.05 mm Mono-capillay 135 * 0.05 mm Mono-capillay 135 * 0.5 mm	Pre-gelected products: Hybrid Monochr. 2xGe220 Cu Asym. (MPD) Mirror Cu W/Si (parabolic MPD / Cap.Spin.) Progr. Div. Slit & Anti-scatter Slit <
Mirror Cu W/Si [parabolic MRD) Mono-capillay 135 ° 0.05 mm Mono-capillay 135 ° 0.05 mm Mono-capillay 135 ° 0.3 mm Mono-capillay 135 ° 0.3 mm Mono-capillay 150 ° 0.5 mm Mono-capillay 165 ° 0.05 mm Mono-capillay 155 ° 0.25 mm Mono-capillay 15	
monochromator. For Cu radiation. Creates high intensity parallel beam.	OK Cancel Help
Pre-celested ever	duala.

Pre-selected products:					
Hybrid Monochr. 2xGe220 Cu Asym. (MPD)					
Mirror Cu W/Si (paral	Mirror Cu W/Si (parabolic MPD / Capillary Spinner)				
Progr. Div. Slit & Anti-scatter Slit					
C Offsets					
Default	-1.102				
Lotal:	-1.115				

• 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.

Pre-selected products:				
Gas-flow PSD				
PW3011/20 (Miniprop. large window)				
X'Celerator				
Detector properties				
Number:	1 2 3 4			
Detector offset (mm):	0.5			
_				
Pitch (mm):	0.0539			

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

🚺 Save Co	nfiguratio	on As			
<u>N</u> ame:	My Spinne				ОК
Description:	Configurati	on for Powder Sample	s (QSG)		Cancel
- Existing co	onfigurations	:		5	Help
Name My Other	Optics	Description Example for Other Ar	Date & Time 20-Sep-2004 14:59		
My Spinn My Triple	ar Axis	Configuration for Pov Configuration for Higl	21-Sep-2004 13:37 20-Sep-2004 14:45		
Users Availa	able to all us	ers			
Name	•				
User-	1 ame				



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

Press OK and then X.

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 ...

🚺 New Progr	am	×
<u>P</u> rogram type:	Relative scan 💌	OK Cancel Help

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



3.5.1 Defining the Measuring Program Parameters

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

🚯 Prepare Absolute Scan [Program	m1]		_ 🗆 🛛
Configuration High-throughput Scan Axis Gonic Uther gonic angle Uther gonic angle Uther donic angle at start Offset (*): Uther donic angle at start	Scan properties Repetition Step Continuous Pre-set counts Start angle (*): End angle (*): Step size (*): Time per step (s): Scan speed (*/s): Pre-set counts (counts): Number of steps: Total time (h:m:s):	5.000 70.000 0.020 0.50 0.040000 10000 3250 00:27:08	Comment Settings

If the "Configuration" is not My Spinner, select it from the dropdown 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.

🚯 Prepare Absolute Scan [Program			
Configuration My Spinner Scan Axis Gonio Other gonio angle Uther gonio angle Uthe	Scan properties Repetition Step Continuous Pre-set counts Start angle (*): End angle (*): Step size (*): Time per step (s): Scan speed (*/s): Pre-set counts (counts): Number of steps: Total time (h:m:s):	20.000 90.000 0.020 1.00 0.020000 1.000 3500 0.058:23	Comment Settings

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 <u>Settings...</u> to open a window in which we can specify the actual hardware settings that we are going to use.

🚯 Program1: Settings	×
□	~
Movement = not moving	
🚊 🧛 Incident beam path	
PreFIX module: Actual	
🚽 🕅 📉 📉 🖌 🖌 Mati-scatter slit: Actual	
	Ξ
🚽 🥏 Beam knife: Actual	
🚽 🕅 Divergence slit: Actual	
Filter: Actual	
Mask: Actual	-
🚽 💭 Mirror: Actual	
Monochromator: Actual	
Difference of the second bases and the second bases are the second bases	
Dirracted beam path: Dirracted beam path i ProElX module: Actual	
	_
OK Cancel Apply Help	

• 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".



Program1: Settings	×
Sample stage: Spinner PW3064 Movement = spinning enabled, revolution time: 1.0000 s Lift = Up Incident beam path PreFIX module: Actual Anti-scatter slit: Actual Seam attenuato: Actual	
Beam knife: Actual Divergence slit: Actual Filter: Actual Mask: Actual Mirror: Actual Monochromator: Actual Soller slit: Actual Diffracted beam path1	
Sample stage movement	
Spinner PW3064	
Spinning Revolution time (s): Oscillation Not moving	
OK Cancel Apply Help	כ

• 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").

🚺 Save Pro	ogram As				
<u>N</u> ame:	Name: My Program				
Description:	Example fo	r QSG		Cancel	
- Existing pro	ograms:			Help	
Name		Description	Date & Time		
- Users Availa	able to all us	ers			
Name	•				
User- My N	1 ame				



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

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

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":

ť	Instrument Se	ttings	X
	Position Sample	Stage Sample Changer X-ray	
	X-ray generat	.or	
	<u>T</u> ension (kV):	45 Generator on	
	<u>C</u> urrent (mA):	40	
	X-ray tube		
	∐-ray tube:	PW3373/10 Cu LFF DK137133 💌	
		Breed	
	Shutter		
		Line focus O Point focus	
		Shutter open	
	ок (Cancel Apply Help	

- **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 enclo- sure 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 sam- ple 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 sam- ple 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

• Start the measurement program by selecting Measure - Program ...

🚯 Open Program	
List programs of type: Absolute scan	OK
Name Description Date User My Program Example for Quick Stz[21-Sep-2004-13:5(]My Name	Cancel
	нер

- Highlight your program (in this example: "My Program") and press
- 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

🚯 Start			
Program			
Name:	My Program		
Туре:	Absolute scan		
Description:	Example for Quick Start Guide		
File			
<u>N</u> ame:	My Program_1.xrdml		
<u>F</u> older:	C:\X'Pert Data\My Name		
<u>C</u> omment:	Example for Quick Start Guide		
Sample			
<u>I</u> D:	Standard Si Sample		
<u>N</u> ame:	Silicon Pellet		
Prepared by:	My Full Name		
Position			
Diffractometer			
_ineta[j:	20.000 Eni (): X (mm):		
Ulfse <u>t</u> (*):			
0_mega (*):	10.000		
Reflection			
Unit cell:	h <u>k</u> l:		
	OK Cancel Help		

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.

ОK

X'Pert Data Collector	
 A Please insert the Incident PreFIX module Progr. Div. Slit ,Anti-scatter Slit (current: None). A Please insert the Incident Soller slit Soller 0.04 rad. (current: None). A Please insert the Incident Mask Inc. Mask Fixed 10 mm (MPD/MRD) (current: None). A Please insert the Diffracted PreFIX module Programmable Receiving Slit (current: None). A Please remove the Diffracted Soller slit Soller 0.02 rad. and insert the Soller 0.04 rad A Please remove the Detector Gas-flow PSD and insert the PW3011/20 (Miniprop. large window). A Please remove the Diffracted Anti-scatter slit Prog. AS Slit and insert the Slit Fixed 1*. 	OK Cancel Ignore

• If there are any actions to be done, perform them and then press

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



Chapter 4

Using X'Pert Data Collector with MRD Type Systems

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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 OK.

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.



1 options				
Diffractometer Hard	Copies Units Miscellane	ous Measuremer	nt Executio	n
Mode				
 Powder 	🔘 Single crystal			
Diffractometer move	ment			
Move diffractome	ter to manual load positions a	after the measurem	ent has fin	ished
Move diffractome	ter automatically when optic	with offset is moun	ted or remo	oved
- Default				
Configuration				
None	*			
		K Ca	ncel	Help

Choose the mode: "Single crystal" and press

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")



🚯 Define Config	uration [Configuration4]		
Configuration	4 tion e family = X'Pert PRO MRD nit: X'Pert PRO n: MPPC • Single system ter: PW3050/65 (Theta/2Theta) tage: None		
System identification			
Identific <u>a</u> tion:			Comment
<u>H</u> ardware family:	X'Pert PRO MRD	*	
<u>C</u> ontrol unit:	X'Pert PR0	~	
<u>G</u> enerator:	MPPC	*	
<u>S</u> ystem:	Single System	*	Previous Next
Read Instrument	Pre-select		

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.

1 Define Configu	ration [Configuration1]			X
Configuration	1 tion = Quick Start Guide configuration 3 e family = X'Pert PR0 MRD nit: X'Pert PR0 : MPPC : Single system ter: PW3050/65 (Theta/2Theta) tage: MRD Cradle beam path lent beam radius = 320.00 mm nded = No			
Take-off angle = 6.0* Shutter port = 1 Diffracted beam path1 Source of the second				>
System identification				
Identific <u>a</u> tion:	Quick Start Guide configuration 3		Comment	
Hardware family:	X'Pert PRO MRD	~		
Control unit: X'Pert PR0				
<u>G</u> enerator:	MPPC	~		
<u>S</u> ystem:	Single System	~	Previous Next	
Read Instrument	Pre-select			

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 0.000°. 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".

1 Define Configu	ration [Configuration4]	
Take Shut Shut Optic Shut Shut Shut Optic Shut Shu	e-off angle = 6.0° ter port = 1 d beam path1 c number = 1 us = 320.00 mm et = 0.000° d beam path2 number = 2 us = 320.00 mm et = 6.000° d beam path3 number = 3 us = 320.00 mm et = 0.000°	
Diffracted beam path	1	
<u>N</u> ame:	Diffracted beam path3	New Delete
R <u>a</u> dius (mm):	320.00	
O <u>f</u> fset (*):	0.0000	
<u>O</u> ptic number:	3	Previous Next
Read Instrument	Pre-select	

Press Delete .

Defining the rest positions

🚹 Manual Load	Positions	×
 ✓ 2Theta (*): ✓ Omega (*): 	0.0000	OK Cancel
✓ Phi (*): ✓ Psi (*):	0.00	
□ × (mm): □ Y (mm): □ Z (mm):		

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.



🚯 Pre-selection			
Image: Pre-selection Product type: PreFIX module (Incident) All products: Mono-capillary 155 * 0.05 mm Mono-capillary 165 * 0.05 mm Mono-capillary 165 * 0.05 mm Mono-capillary 165 * 0.05 mm Mono-capillary 165 * 0.05 mm Mono-capillary 165 * 0.05 mm Mono-capillary 165 * 0.05 mm Mono-capillary 165 * 0.05 mm Mono-capillary 165 * 0.05 mm Mono-capillary 165 * 0.05 mm Mono-capillary 108 * 0.05 mm Mono-capillary 200 * 0.05 mm Mono-capillary 230 * 0.1 mm Mono-capillary 230 * 0.1 mm Mono-capillary 230 * 0.1 mm Mono-capillary 230 * 0.1 mm Mono-capillary 230 * 0.1 mm Mono-capillary 230 * 0.1 mm Mono-chromator 4x6e220 Cu Asym. (MR Mono-chromator 4x6e220 Cu Asym. Mirro Monochromator 4x6e220 Cu Asym. Mirro Monochromator 4x6e220 Cu Sym. Mirro Monochromator 4x6e220 Cu S	D) x x (MRD)) eam. For use on r Cu radiation.	Pre-selected products: Mirror Cu W/Si [parabolic MRD) Monochromstor 4xGe220 Cu Sym. Mirror IM Progr. Div. Silt & Anti-scatter Silt Default: 0.0000 Iotal: 0.0000 Iotal: 0.0000	PD

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.

Pre-selected products:					
Mirror Cu W/Si (parabolic I	Mirror Cu W/Si (parabolic MRD)				
Monochromator 4xGe220	Cu Sym. (MRD)				
Progr. Div. Slit & Anti-scatt	er Slit				
C Offsets					
Default:	Default: 0.8817				
<u>I</u> otal: 0.7587					

• 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 \square .

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.

Pre- <u>s</u> elected products:			
PW3011/20 (Miniprop. large window)			
X'Celerator Scientific			
Detector properties			
Number:			
	0.00		
Detector offset (mm):	0.00		
Ritch (mm):			
<u>Licen (ning</u>			

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



🔥 Save Con	figuratio	on As			
<u>N</u> ame: N	Name: My Triple Axis				
Description:	Configurati	on for High resolution	for QSG	Cancel	
Existing con	figurations	:		Help	
Name My Capillar	Spinner	Description Configuration for Car	Date & Time 21-Sep-2004 16:03		
My MPD S My Spinner	My MPD Spinner Configuration for Pov 21-Sep-2004 15:54 My Spinner Configuration for Pov 21-Sep-2004 13:37				
My Triple A	My Triple Axis Configuration for Higl 22-Sep-2004 12:27				
Users Availab	le to all us	ers			
Name User-1 V My Nar	me				

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 **Flead 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"

🚯 Define Config	uration [Configuration3]			
Configuration	3 ation = INTERNAL e family = X'Pert PRO MRD init: X'Pert PRO		^	
Generator: MPPC System = Single system Goniometer: PW3050/65 (Theta/2Theta) Sample stage: MRD Cradle Incident beam path Lincident beam radius = 320.00 mm Extended = No Take-off angle = 6.0* Shutter port = 1				
- 🏎 Opti - 👞 Rad	c number = 1 ius = 320.00 mm		~	
CDiffracted beam pat	h			
<u>N</u> ame:	Diffracted beam path1	*	New Delete	
R <u>a</u> dius (mm):	320.00			
O <u>f</u> fset (*):	0.0000			
<u>O</u> ptic number:	1		Previous Next	
Read Instrument	Pre-select			

and remove it from the configuration by pressing Delete.

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



🚹 Define Config	uration [Configuration3]	
Configuration Hardwa Control of Generat System Sample Sample Incident Exter Star Diffracte Optimate Optimate Optimate Optimate	a3 ation = INTERNAL e family = X'Pert PRO MRD unit: X'Pert PRO or. MPPC = Single system ater: PW3050/65 (Theta/2Theta) stage: MRD Cradle beam path dent beam radius = 320.00 mm ended = No e-off angle = 6.0° tter port = 1 d beam path3 c number = 3 iius = 320.00 mm	
Diffracted beam pat	h	
<u>N</u> ame:	Diffracted beam path3	New Delete
R <u>a</u> dius (mm):	320.00	
O <u>f</u> fset (*):	0.0000	
Optic number:	3	Previous Next
Read Instrument	Pre-select	

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:

🚯 Manual Load Positions				
 ✓ 2Theta (*): ✓ Omega (*): 	0.0000	OK Cancel		
 Phi (*): Psi (*): 	0.00			
□ × (mm): □ Y (mm): □ Z (mm):				

- 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.



1 Pre-selection	X
Product type: PreFIX module (Incident) All products: Mono-capillary 150 * 0.5 mm Mono-capillary 155 * 0.05 mm Mono-capillary 155 * 0.5 mm Mono-capillary 155 * 0.05 mm Mono-capillary 155 * 0.05 mm Mono-capillary 230 * 0.1 mm Mono-chromator 4x6e20 Cu Asym. MIRD1 Monochromator 4x6e20 Cu Asym. MIRD1 Monochroma	Pre-gelected products: Mirror Cu W/Si [parabolic MRD] Monochromator 4x6e220 Cu Sym. Mirror (MRD) Progr. Div. Silt & Antiscatter Sit <
	OK Cancel Help

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.

Pre-selected products:				
Mirror Cu W/Si (parabolic	MRD)			
Monochromator 4xGe220	Cu Sym. (MRD)			
Progr. Div. Slit & Anti-scat	ter Slit			
J				
Offsets				
Default:	0.8817			
Total: 0.7587				

• 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.

Pre-selected products:					
PW3011/20 (Miniprop. large window)					
X'Celerator Scientific					
Detector properties					
Number:	□1 □2 ☑3 □4				
Detector offset (mm):	0.00				
D'elector onset (mm).					
Pitch (mm):					

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



🔞 Save Configuration As					
<u>N</u> ame:	Mt Other C	Mt Other Optics			
Description:	Configurati	ion for other analyses I	for QSG	Cancel	
∠Existing co	onfigurations	c		Help	
Name		Description	Date & Time		
My Capilla My MPD :	ary Spinner Spinner	Configuration for Cap Configuration for Pov	21-Sep-2004 16:03 21-Sep-2004 15:54		
My Spinn My Triple	My Spinner Configuration for Pov 21-Sep-2004 13:37 My Triple Axis Configuration for Higl 22-Sep-2004 12:27				
- Users Availa	Users Users Available to all users				
Name					
User-1 W Name					
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")

🚯 Connect				\mathbf{X}
Configuration Name Mt Other Optics My Capillary Spinner My MPD Spinner My Spinner My Spinner My Triple Axis	Description Configuration for other Configuration for Capilla Configuration for Powd Configuration for Powd Configuration for High	Date 22-Sep-2 21-Sep-2 21-Sep-2 21-Sep-2 22-Sep-2	Dwner My Name My Name My Name My Name My Name	OK Cancel Help
Diffracted Beam Paths O Diffracted beam path1 O 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.



🔥 Incident beam path: Assuming radius: 320 mm	OK
Please remove the current Incident Beam attenuator.	
A Incident Monochromator. Assuming 4xGe440 Cu Sym	Cancel
A Please remove the current Incident Mask.	
A Please remove the current Divergence slit.	
🔥 Diffracted beam path1: Diffracted beam path1: Assuming radius: 320 mm	
Diffracted beam path1: Diffracted Monochromator. Assuming Triple axis, 3xGe220 Cu Sym	
A Diffracted beam path1: Please remove the current Diffracted Beam attenuator.	
<u> Diffracted beam path</u> 2: Diffracted beam path2: Assuming radius: 320 mm	
A Diffracted beam path2: Please remove the current Diffracted Beam attenuator.	
,	

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 **DK** 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 Se	ettings 🛛 🔀
Position Sample	Stage X-ray
X-ray general	tor
<u>⊺</u> ension (kV):	45 Generator on
<u>C</u> urrent (mA):	40
X-ray tube	
⊠-ray tube:	PW3373/10 Cu LFF DK137618 🔽
	Breed
Shutter	
	Shutter open
ОК	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"

My Triple Axis			×
Instrument Settings	Incident Beam Optics	Diffracted Beam Op	tics
E States Incident b Radius	e am path = 320.00 mm ff angle = 6.0000 *		
1 Incident	Beam Optics		×
Monochro PreFIX M <u>I</u> ype:	imator Beam Attenu lodule Divergenc	iator Filter e Slit Mask	Beam Knife
ОК	Cancel	Apply	Help

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.



	Cancel
--	--------

- Carry on selecting the items in the beam path by selecting the relevant
- tabs and the types from the drop-down lists and pressing
- 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".

My Triple Axis		×				
Instrument Settings Incident B	eam Optics	Diffracted Beam Optics				
Diffracted beam path1 Diffracted beam path1	h2					
Optic number = 2						
- 🕰 Radius = 320.00 mm						
Vetector: PW3011/20 (Miniprop. large window)[2]						
PHD lower lev	el = 33.0% el = 80.0%					
Factory PHD Ic	wer level = 2	20.0%				
	pper lever =					
	🔉 Diffraci	ted Beam Optics				
	Soller Slit	Monochromator Collin	nator Detector E	Beam Attenuator		
	PreFIX M	odule Anti-scatter Slit	Receiving Slit	Filter Mask		
	Ŧ					
	The:	None		<u> </u>		
	OK	Cancel	Apply	Help		

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.


Please insert the Diffracted PreFIX module Triple axis (Rocking	OK
curve optics) (current: None).	Cancel

- Perform the actions as instructed and press _____K.
- 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.

My Triple Axis	×				
Instrument Settings Incident Beam Optics Diffracted Beam Optics					
E- 😃 Diffractometer					
🗄 🦓 Positions	🖪 Instrument Settings 🛛 🔀				
Offset = 0.0000*	Position Sample Stage X-ray				
- Rev Omega = 14.2210*					
Phi = 0.00*	<u>2</u> Theta (*): 5.0000 ≚ (mm): 0.00 🛨				
→ Psi = 0.00°	O <u>f</u> fset (*): 0.0000 ⊻ (mm): 0.00 ÷				
	0				
	<u>U</u> mega(): 2.5000 ≧(mm): 0.000 -				
🖻 🙀 Goniometer: PW3050/65 (Theta/2Theta)	Phi(*): 0.00 Unit cell: None 🔽				
Secola stass: MPD Code	Prift 0.00 bkt				
Sample mode = Beflection	12().				
Movement = not moving	Move to Manual Loading Position				
E- 🐪 X-ray	Goniometer				
Generator: MPPC	Besolution: High (0.0001)				
🛛 🛶 Status = On					
Tension = 45 kV					
🛶 🕰 Current = 40 mA					
- 🌺 X-ray tube: PW3378/00 Cr LFF DK969348					
🖃 💥 Shutter					
Status = Closed					
Focus = Line focus					
······································	OK Cancel Apply Help				

- To change the positions: enter the new value and press
- 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 ($Psi = 0^\circ$), 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".

🚯 Prepare Manual	Scan			
Scan axis				Start Save As
Scan mode	Scan parameters			
🔘 Step	Range (*):	0.9900	Pre-set counts (counts):	10000
 Continuous 	Step size (*):	0.0100	Number of steps:	99
O Pre-set counts	Time per step (s):	0.50	Total time (h:m:s):	00:00:53
	Scan speed (*/s):	0.020000		

- In the instrument control window "Instrument Settings" tab, doubleclick 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 <u>Start</u> 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.



Optimize Psi 4.5.6

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 2 nd axis, which is the F	si axis, set:	

Range:	4
Step size:	0.5

🚯 Prepare Optimize	Program [Pr	ogram2]		
Optimize mode Stop at level Go to maximum Go to maximum 1st o Optimize Phi Optimize Psi	derivative	Configuration My Triple Axis Diffracted Beam Paths Diffracted beam pa O Diffracted beam pa	► th1 th2	Comment Settings
Range (*): Step size (*): Time per step (s): Scan speed (*/s): Stop level (cps): Number of steps: Time (h:m:s):	0.0900 0.0100 0.50 0.020000 1000 100 00.00.08	Scan Axis Omega 2nd axis Psi Range (*): Step size (*): Number of scans: Total time (h:m:s):	 4.00 0.50 41 00:08:20 	

- 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.

🚯 Save Program As				
Name: My Optimize Program			ОК	
<u>D</u> escription:	Example fo	r Quick Start Guide		Cancel
Existing pro	ograms:			Help
Existing programs: Name Description Date & Time My Capillary Program Example for Quick SI 21-Sep-2004 16:06 My Monochromator F Example for Quick SI 22-Sep-2004 09:34 My Program Example for Quick SI 21-Sep-2004 13:50 Users Available to all users Name User-1 W Name My Name				

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



4.5.6.2 **Optimizing the Sample Orientation**

• Select Measure - Program...

🚯 Open Program			
List programs of type:	Optimize program	~	
Name My Optimize Program	Description Example for Quick Start 0	Date Use iuide 22-Sep-2004 My f	Cancel Help

- 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.

🔥 Start	×				
Program					
Name:	My Optimize Program				
Туре:	Optimize program				
Description:	Example for Quick Start Guide				
File					
<u>N</u> ame:	My Optimize Program_1.xrdml				
<u>F</u> older:	C:\X'Pert Data\My Name				
<u>C</u> omment:	Example for Quick Start Guide				
Sample					
<u>I</u> D:	Si(111) Sample				
<u>N</u> ame:	Standard High Resolution Sample				
Prepared by:	My Full Name 🔍				
Position					
Diffractomete					
<u>2</u> Theta (*):	28.4410 Phi (*): 0.00 ≚ (mm): 0.00				
Offse <u>t</u> (*):	0.0089 Psi (*): 0.00 Y (mm): 0.00				
<u>O</u> mega (*):	14.2294 ∠ (mm): 7.226				
-Reflection-	Reflection				
<u>U</u> nit cell:	Si[111]				

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.

Omega: 14.2068 2Theta: 28.4410 Phi: 0.00 Psi: -1.50 Count rate: 16.0

• 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.

🚯 New Progr	am	X
Program type:	Relative scan	OK Cancel
		Help

- 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

🚺 Prepare Relative Scan [Program	n2]		
Configuration	Scan properties Repetitio	n	
My Triple Axis 🔽	🔿 Step		Comment
Diffracted Beam Paths	 Continuous 		Settings
 Diffracted beam path1 Diffracted beam path2 	Pre-set counts		
	Range (*):	0.1890	
Scan axis	Step size (*):	0.0010	
Umega	Time per step (s):	0.50	
	Scan speed (*/s):	0.020000	
	Pre-set counts (counts):	10000	
	Number of steps:	189	
	Total time (h:m:s):	00:01:40	

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

🚯 Save Pro	🗴 Save Program As				
<u>N</u> ame:	Name: My Rocking Curve Program				
Description:	Example fo	r Quick Start Guide			Cancel
Existing pro	ograms:				Help
Name My Capilla My Monoo My Optimi My Progra	ry Program chromator F ze Program m	Description Example for Quick SI Example for Quick SI Example for Quick SI Example for Quick SI	Date & Time 21-Sep-2004 16:06 22-Sep-2004 09:34 22-Sep-2004 15:11 21-Sep-2004 13:50		
Users Availa User- User- My Na	ble to all us : 1 ame	ers			



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

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.

🚯 Start	Σ	K			
Program					
Name:	My Rocking Curve Program				
Туре:	Relative scan				
Description:	Example for Quick Start Guide				
File		Š.			
<u>N</u> ame:	My Rocking Curve Program_1.xrdml				
<u>F</u> older:	C:\X'Pert Data\My Name				
<u>C</u> omment:	Example for Quick Start Guide				
	×				
Sample		ĥ			
ID:	Si(111) Sample				
<u>N</u> ame:	Standard High Resolution Sample				
Prepared by:	My Full Name				
Position		ĥ			
Diffractometer					
<u>2</u> Theta (*):	28.4410 Ehi (*): 0.00 ≚ (mm): 0.00				
Offset (*):	0.0089 P₂i (*): 0.34 ⊻ (mm): 0.00				
<u>O</u> mega (°):	14.2294 <u>∠</u> (mm): 7.226				
Reflection					
<u>U</u> nit cell: Si	i[111] ▼ h <u>k</u> l: 111				
	OK Cancel Help	J			

• 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



4.6 PERFORMING A REFLECTIVITY MEASUREMENT4.6.1 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.

🚯 Connect					
Configuration					JK
Name	Description	Date	Owner		naal
Mt Other Optics My Capillary Spinner My MPD Spinner My Spinner	Configuration for other Configuration for Capilla Configuration for Powd Configuration for Powd	22-Sep-2 21-Sep-2 21-Sep-2 21-Sep-2	My Name My Name My Name My Name	н	lelp
My Triple Axis	Configuration for High	22-Sep-2	My Name		
Diffracted Beam Paths O Diffracted beam path1					
 Diffracted beam path2 					

• Select "My Other Optics" in the "Configuration" frame and press

DK. 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:

🖄 Instrument S	ettings	
Position Sample	e Stage X-ray	
X-ray genera	itor	
<u>T</u> ension (kV):	45 Generator on	
<u>C</u> urrent (mA):	40	
X-ray tube		
∐-ray tube:	PW3373/10 Cu LFF DK137618 💌	
	Breed	
Shutter		
	Line focus Point focus Shutter open	
ОК	Cancel Apply He	elp

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



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".

Mt Other Optics		×	<
Instrument Settings	Incident Beam Optics	Diffracted Beam Optics	🛚 🚺 Incident Beam Optics
Incident b Radius Radius Redius Re	eam path = 320.00 mm f angle = 2.4200 * module: Monochromator set = 0.0000* nochromator: 4x6e220 C sk: Crossed Silt Collimato Width = 1.00 mm ergence silt: Crossed Silt Height = 1.00 mm nc. Beam Cu W/Si (MEI set = 0.881** ttenuator: Ni 0.125 mm a stot = 20.00 scription = Software defa age = Do not switch tus = De-activated	4xGe220 Cu Sym. Mirror Cu Sym. (mirror) xr (MRD) : Collimator (MRD) D) sutomatic ult Beam Attenuation Fac	Monochromator Beam Attenuator Filter Soller Silt Beam Knife PreFIX Module Divergence Silt Anti-scatter Silt Mask Mirror Iype: Monochromator 4xGe220 Cu Sym. Mirror (MRD) Default offset (*): 0.0000 Iotal offset (*): 0.0000
			OK Cancel Apply Help

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

If you get a message telling you that you have actions to perform, do the actions as instructed and press $\Box CK$.

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



X'Pert Data Collector				
▲ Set incident beam mask width to 10.00. ▲ Please confirm that the mirror is used in Extended position.	OK Cancel			

• Carry out the requested actions and then press **DK**. 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.



My Other Optics	×
Instrument Settings Incident Beam Optics Diffrac	cted Beam Optics
Instrument Settings Incident Beam Uptics Diffracted beam path3 © Diffracted beam path3 © Optic number = 1 PR Radius = 320.00 mm PR Used wavelength = K-Alpha1 [= 2.2897 © Detector: XCelerator Scientific[3] PR Mode = Receiving slit PR Mode = R	7600 Å)
	OK Cancel Apply Help
Press Apply X'Pert Data Collector Please insert the Diffracte 0.27* (current: None).	d PreFIX module Parallel Plate Collimator

- Perform the actions as instructed and press
- 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





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.



🚯 Instrumen	t Settings			X
Position Sa	mple Stage X-ra	y]		
<u>2</u> Theta (*):	0.0000	∐ (mm):	0.00	
0 <u>f</u> fset (*):	0.0000	⊻ (mm):	0.00	
<u>O</u> mega (*):	0.0000	<u> ∠</u> (mm):	7.226	
<u>P</u> hi (°):	0.00	<u>U</u> nit cell:	None	~
P <u>s</u> i (*):	90.00	h <u>k</u> l:		
		Move to Manu	al Loading Position)
Goniomet	er			
<u>R</u> esolution:	High (0.0001)		*	
OK Cancel Apply Help				

• 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.

Mt Other Optics X					
Instrument Settings	Incident Beam Optics	Diffracted Beam Optics			
Incident I Radius Radius Takeol Off Off Mo Off Mirror: I Pos Off Off	eam path = 426.70 mm if angle = 7.0000 ° module: Monochromator set = 0.0000° nochromator: 4xGe220 0 sk: Crossed Slit Collimato Width = 10.00 mm ergence slit: Crossed Slit Height = 1.00 mm nc. Beam Cu W/Si (MRI sition = extended set = 0.0000° ttenuator: NI 0.125 mm a ctor = 165.90 scription = X'Pert PRO M age = Do not switch tus = Activated	4xGe220 Cu Sym. Mirror Cu Sym. (mirror) r (MRD) : Collimator (MRD) D) sutomatic RD			

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.



🔥 Status Bar				
ltem <u>1</u>	Item 2	Item <u>3</u>	ltem <u>4</u>	Item 5
None Onego 21heta Phi Psi X PDS divergence PDS irradlated length PDS offset PASS scatter PASS observed length PASS offset PRS height	None Omega 2Theta Phi Psi X Z PDS divergence PDS irradiated length PDS offset PASS scatter PASS observed length PASS offset PRS height	None Dmega 2Theta Phi Psi X Y PDS divergence PDS firadiated length PDS offset PASS coatter PASS coatter PASS observed length PASS offset	 None Omega ZTheta Phi Pi X Y Z PDS divergence PDS diset PASS scatter PASS observed length PASS breat PRS height 	Phi Phi Pi Pi Pi Z PDS divergence PDS invadiated length PASS observed length PASS observed length PASS observed length PBS height Beam att. Spinore setting Count rate

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

		Save do
Scan parameters		
Range (*):	0.9900	Pre-set counts (counts): 1000
Step size (*):	0.0100	Number of steps: 9
Time per step (s):	0.50	Total time (h:m:s): 00:00:5
Scan speed (*/s):	0.020000	
	Scan parameters Range (*): Step size (*): Time per step (s): Scan speed (*/s):	Scan parameters Range ('): 0.9900 Step size ('): 0.0100 Time per step (s): 0.50 Scan speed ('/s): 0.020000

- 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 \boxtimes 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 \Rightarrow button on the toolbar.
- Double-click on the Beam Attenuator branch of the tree view.

ľ,	Incident Beam	Optics		X
	PreFIX Module D Monochromator B	ivergence Slit eam Attenuato	Anti-scatter Sl Filter Solle	it Mask Mirror erSlit Beam Knife
	<u>Type:</u> Ni 0.125	mm automatic		~
	Attenuation factor	:	165.90	Select
	<u>U</u> sage:		Do not switch	~
			Activated	
	ОК	Cancel	Apply	Help

- 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
- Select *System Settings Beam Attenuation Factors* press **New** and define the attenuation factor for your attenuator (in this example: "165.9").



Name	Description	Factor	ОК
Ni 0.125 mm automatic Ni 0.125 mm automatic	Software Default Beam Attenuation X'Pert PB0 MBD	20.00	New
			Modify
			Delete
			Print
			Help

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

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

🚺 Prepare Manual	🛿 Prepare Manual Scan						
Scan axis				Start			
2Theta	✓			Save As			
Scan mode	Scan parameters						
🔿 Step	Range (*):	0.9900	Pre-set counts (counts):	10000			
 Continuous 	Step size (*):	0.0100	Number of steps:	99			
Pre-set counts	Time per step (s):	0.50	Total time (h:m:s):	00:00:55			
	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.
- 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 (\checkmark) "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

 Select Measure – Manual Scan and then on the "Prepare Manual Scan" window select "Omega" and press Statt.

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.

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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).

🚯 Sample C	X		
	Current position	Sample offset	
<u>2</u> Theta (*):	0.0000	0.0101	Set New = 0
<u>O</u> mega (*):	0.0000	-0.0670	Clear All Offsets
<u>P</u> hi (*):	0.00	0.00	
P <u>s</u> i (*):	0.00	0.00	
∐ (mm):	0.00	0.00	
⊻ (mm):	0.00	0.00	
<u>Z</u> (mm):	7.830	0.000	
	ОК	Cancel	Help

- 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
- Enter the following parameters:

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

Configuration Scan properties Repetition Comment My Other Optics Step Settings Scan Axis Continuous Pre-set counts Other gonio angle Use actual angle at start Step ize (*): 0.0050 Offset (*): Image of the set of	🔀 Prepare Absolute Scan [Program	m4]		
Scan speed (*/s): 0.001000 Pre-set counts (counts): 10000 Number of steps: 240 Total time (h:m:s): 00:20:08	Configuration My Other Optics	Scan properties Repetition Step Continuous Pre-set counts Start angle (*): End angle (*): Step size (*): Time per step (s): Scan speed (*/s): Pre-set counts (counts): Number of steps: Total time (h:m:s):	on 0.3000 1.5000 0.0050 5.00 0.001000 10000 240 00.20:08	Comment Settings

- 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.

🚯 Program 7: Settings		X				
🖃 🔀 Sample stage: MBD Cr	iradle	~				
Movement = not m	novina					
🖃 🧟 Incident beam path						
PreFIX module: Actual						
Anti-scatter slit: Actual						
😑 🍻 Beam attenuator: N	Ni 0.125 mm automatic					
	90					
→ Description = >	X'Pert PRO MRD					
Usage = At pre	e-set intensity					
Activate level	= 500000 cps					
Beam knife: Actua	svei = 400000 cps					
Divergence slit: Ac	ctual					
Filter Actual						
Mask: Actual						
Incident Beam attenuator						
Ni 0.125 mm automatic	×					
Attenuation factor:	165.90 Select					
<u>U</u> sage:	At pre-set intensity					
Activate level (cps):	500000					
De-activate level (cps): 200000						
ОК	Cancel Apply Help					
		_				

- 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 X.



4.6.7 Measuring the Reflectivity Curve

- Select Measure Program and press
- 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.

Program					
Name:	My Reflec	tivity Progra:	m		
Туре:	Absolute :	scan			
Description:	Example f	or Quick Sta	art Guide		
File					
<u>N</u> ame:	My Reflec	My Reflectivity Program_1.xrdml			
<u>F</u> older:	C:\X'Pert	C:WPert Data\My Name			
<u>C</u> omment:	Example f	or Quick Sta	art Guide		~
					~
Sample					
D:	Thin Film	Thin Film Sample			
<u>N</u> ame:	Cr Layer				
Prepared by:	My Full Na	ame			
Position					
-Diffractometer					
2Theta (*):	0.3000	<u>P</u> hi (*):	0.00	∐ (mm):	0.00
Offse <u>t</u> (*):	0.0000	P <u>s</u> i (*):	0.00	⊻ (mm):	0.00
<u>O</u> mega (*):	0.1500			<u>Z</u> (mm):	7.830
Reflection					
<u>U</u> nit cell:			h <u>k</u> I :		
			ок 🗌 🚺	Cancel	Help

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....

	🔥 Axes			
	Intensity : 10 20 50 100 200 500 1 k 2 k	scale:	Units © Counts © cps Type © Linear © Square root Cogarithmic	Close Help
	Auton Auton Auton Show Show X-axis Center	natic change o matic X-scaling markers intermediate ro in Arcsecs er 0.9	f intensity scale esults	
Select:	Units: Type: Check	Counts Logarith "Autom	umic atic change of	intensity scale
	Check	"Autom	atic X-scaling	"

Press Close

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

٠





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


Chapter 5

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

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5.1 INTRODUCTION

In this chapter we are going to perform two measurements:

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

5.1.1 System

The system that we will use to gather the data is an X'Pert PRO 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
 Collector
- Enter the user name and password: "My Name" and "password" and press OK.

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).

🕻 Define Configuration [Configuration1]			
Configuration1 Addware family = XPert PR0 MPD Control unit: XPert PR0 Control unit: XPert PR0 System = Single system Coniometer: None Sample stage: None			
System identification			
Identific <u>a</u> tion:		Comment	
<u>H</u> ardware family:	X'Pert PRO MPD	~	
<u>C</u> ontrol unit:	X'Pert PR0	*	
<u>G</u> enerator:	MPPC	∽	
<u>S</u> ystem:	Single System	Previous Next	
Read Instrument	Pre-select		

 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.

🚺 Define Config	uration [Configuration1]			
Configuration1 Identification = Intern Demo App Lab Hardware family = X'Pert PR0 MPD Control unit: X'Pert PR0 Generator: MPPC System = Single system Goniometer: PW3050/60 (Theta/2Theta) Sample stage: Spinner PW3064 Sample changer: Changer PW3065/00 (15 positions) Incident beam path Katended = No Katended = No				
System identification	I			
Identific <u>a</u> tion:	Intern Demo App Lab	Comment		
Hardware family:	X'Pert PRO MPD			
Control unit: X'Pert PRO 🗸				
Generator: MPPC 💌				
System: Single System Vervious Next				
Read Instrument	Pre-select			

• 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.

🚺 Define Configuration [Configuration1]	
Configuration1 Configuration1 Configuration1 Control unit: X'Pert PR0 MPD Control unit: X'Pert PR0 Control unit: X'Pert P	
Incident beam path	
Radius (mm): 240.00 Extended radius (mm):	
Shutter port: 1	
Previous 1	Vext
Read Instrument Pre-select	

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

 Diffracted beam pat 	h	
<u>N</u> ame:	Diffracted beam path1	New Delete
	-	
Radius (mm):	240.00	
Offset (*):	0.000	
-2()/		
Optic number:	1	Previous Next
optic number.		TICYIOUS TICK

• 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.

🚯 Pre-selection	
Deskethers D. Dir. 11 (1, 11, 0)	
Product gype: PreFix module (incident)	
All products:	Pre-selected products:
Fixed Collimator Point Focus	
Fixed Div. Slit & Anti-scatter Slit	
Fixed Divergence Silt Hubrid Managha 29G a220 Ca Asum (MPD)	
Hubrid Monochi, 2xGe220 Cu Asym, (MPD) Hubrid Monochi, 2xGe220 Cu Asym, (Cap Spin /MPD	
Hybrid Monochr. 2xGe220 Cu Asym. (Cap.Spin./MRC	
Hybrid Monochr. 2xGe220 Cu Asym. (MPD)	
Hybrid Monochr. 4xGe220 Cu Sym. (MPD)	
Hybrid Monochr. Ni/C 4xGe220 Cu Sym. (MPD)	Offsets
Mirror Co W/Si (focusing MPD / Cap.Spin.)	Default 0
Mirror Co W/Si (focusing MPD)	
Mirror Co W/Si (parabolic MPD / Cap.Spin.)	
Mirror Lo W/Si (parabolic MPU) Mirror Cu) (/Si (focusing MPD / Cop Spin)	
Minor Cu W/Si (focusing MPD / Cap.spin.)	
Mirror Cu W/Si (narabolic MPD / Can Spin)	
Mirror Cu W/Si (parabolic MPD)	
Mirror Cu W/Si (parabolic MRD / Cap.Spin.)	
Mirror Cu W/Si (parabolic MRD)	
Mono-capillary 135 * 0.05 mm	
Mono-capillary 135 * 0.1 mm	
Mono-capillary 135 * 0.3 mm	
Mono-capillary 135 ° U.5 mm	
Mono-capillary 150 0.5 mm	
Mono-capillary 165 * 0.01 mm	Select Product
Mono-capillary 165 * 0.05 mm	
	-
Select Product	

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.

TA Pre-selection	×
Pre-selection Product type: PreFIX module (Incident)	Pre-gelected products: Pre-gelected products: Hybrid Monochr. 2xGe220 Cu Asym. (MPD) Mirror Cu W/Si (parabolic MPD / Cap.Spin.) Progr. Div. Slit & Anti-scatter Slit
Mirror Co W/Si (parabolic MPD) Mirror Cu W/Si (focusing MPD / Cap.Spin.) Mirror Cu W/Si (focusing MPD) Mirror Cu W/Si (parabolic MPD) Mirror Cu W/Si (parabolic MPD) / Cap.Spin.) Mirror Cu W/Si (parabolic MPD) Mono-capillary 135 ° 0.05 mm Mono-capillary 135 ° 0.05 mm Mono-capillary 135 ° 0.5 mm Mono-capillary 135 ° 0.5 mm Mono-capillary 150 ° 0.3 mm Mono-capillary 150 ° 0.5 mm Mono-capillary 155 ° 0.05 mm Mono-capillary 155 ° 0.05 mm	Iotat 3.305
Mono-capillary 165 * 0.2 mm Mono-capillary 165 * 0.2 mm PW3149/63. Incident beam. Contains Mirror W/Si [parabolic MPD] and flat 2-bounce Ge 220 asymmetrical monochromator. For Cu radiation. Creates high intensity parallel beam.	OK Cancel Help
Pre- <u>s</u> elected prod Hybrid Monoch. Mirror Cu W/Si (Progr. Diy, Slit &	lucts: 2xGe220 Cu Asym. (MPD) parabolic MPD / Capillary Spinner] Antiscatter Slit

- Hybrid Monochr. 2x6e220 Cu Asym. (MPD) Mirror Cu W/Si (parabolic MPD / Capillary Spinner) Progr. Div. Slit & Anti-scatter Slit Offsets Default: -1.102 Iotal: -1.110
- 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.

Pre-selected products:				
Gas-flow PSD				
PW3011/20 (Miniprop. k	arge window)			
X'Celerator				
Detector properties				
Number:	1 2 ⊻3 4			
Detector offset (mm):	0.5			
Pitch (mm):	0.0539			
<u>r</u> .con (min):	0.0000			

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

🕼 Save Configuration As	
Name: My MPD \$pinner	ОК
Description: Configuration for Powder Samples (QSG)	Cancel
Existing configurations:	Help
Name Description Date & Time My Other Optics Example for Other Ar 20-Sep-2004 14:59 My Spinner Configuration for Pow 21-Sep-2004 13:37 My Triple Axis Configuration for Higl 20-Sep-2004 13:37 My Triple Axis Configuration for Higl 20-Sep-2004 14:45 Users Available to all users User-1 My Name	

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.

🚯 Save Configuration As					
<u>N</u> ame: <u>My Capi</u>	Name: My Capillary Spinner				
Description: Configur	ation for Capillary Sampl	es (QSG)	Cancel		
Existing configuratio	ns:		Help		
Name	Description	Date & Time]		
Mt Other Optics	Configuration for othe	22-Sep-2004 15:46]		
My Capillary Spinne	r Configuration for Cap	21-Sep-2004 16:03			
My MPD Spinner	Configuration for Pov	21-Sep-2004 15:54			
My Spinner	Configuration for Pov	21-Sep-2004 13:37			
My Triple Axis	Configuration for Hig	22-Sep-2004 14:11			
Users					
Name					
My Name	My Name				

- 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.

1 New Progr	am	
<u>P</u> rogram type:	Absolute scan	🗸 💽
		Cancel
		Help

- 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

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

🚯 Prepare Absolute Scan [Progra	am5]		
A Prepare Absolute Scan Progra Configuration My MPD Spinner Scan Axis Gonio Uther gonio angle Use actual angle at start Offset (*):	Scan properties Repetitio Step Continuous Pre-set counts Start angle (*): End angle (*): Step size (*): Time per step (s): Scan speed (*/s): Pre-set counts (counts): Number of steps: Total time (h:m:s):	28.000 29.000 0.020 10.00 0.002000 10000 50 00.08:23	Comment Settings

- 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
- 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 (°2Theta):" 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.

Press

OK

• Choose the correct configuration and, if applicable, the required beam path.

- Continuation
Name Description Date Owner Mt Other Optics Configuration for other 22-Sep-2 My Name My Capillary Spinner Configuration for Capilla 21-Sep-2 My Name My Spinner Configuration for Powd 21-Sep-2 My Name My Spinner Configuration for Powd 21-Sep-2 My Name My Triple Axis Configuration for High 22-Sep-2 My Name

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If you get any system instructions follow them.

My MPD Spinner		×
Instrument Settings	Incident Beam Optics	Diffracted Beam Optics
Oiffractom Position Position Position Off Off	eter s set = 28.441* set = 0.001* ega = 14.221* eter: PW3050/60 (Thet. solution = Normal (0.001 stage: Spinner PW3064 spinner PW3064 Reflection vement = not moving = Up up herator: MPPC Status = On Tension = 30 kV Current = 10 mA vube: PW3373/00 Cutter Status = Closed Focus = Line focus Port = 1	a/Theta) *) \$

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



🚯 Instrument Se	ttings	
Position Sample	Stage Sample Changer X-ray	
X-ray generat	or	
<u>T</u> ension (kV):	45 Generator on	
<u>C</u> urrent (mA):	40	
X-ray tube		
∐-ray tube:	PW3373/10 Cu LFF DK137133 💟	
	Breed	
Shutter		
	Line focus O Point focus Shutter open	
ок (Cancel Apply	Help

- 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 $\Box CK$.
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	Select the "Sample Stage" tab. Uncheck "Lift Up",
sample changer,	close the doors and press Apply . Open the doors,
but it is positioned	mount the sample, close the enclosure doors, check
in the corner:	"Lift Up" and press OK.
If you have a	Select the "Sample Changer" tab. Open the doors, put
sample changer,	the sample into an empty magazine or monitor position.
and it is ready to	Close the doors, indicate to load the sample from the
use:	position vou just loaded the sample into and press

- 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

 Incident Beam Optics

 PreFIX Module
 Divergence Slit
 Anti-scatter Slit
 Mask
 Mirror

 Monochromator
 Beam Attenuator
 Filter
 Soller Slit
 Beam Knife

 Lype:
 Soller 0.02 rad.

 DK
 Cancel
 Apply
 Help

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 ______ 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".

🚯 Open Program				
List programs of type: Absolu	ite scan		*	ОК
Name	Description	Date	User	Cancel
My Capillary Program	Example for Quick Start Guide	21-Sep-2004	My N	
My Monochromator Program	Example for Quick Start Guide	22-Sep-2004	My N	Нер
My Program	Example for Quick Start Guide	21-Sep-2004	My N	
My Reflectivity Program	Example for Quick Start Guide	23-Sep-2004	My N	
			>	

- 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.



🚯 Start	
Program	
Name:	My Monochromator Program
Туре:	Absolute scan
Description:	Example for Quick Start Guide
File	
<u>N</u> ame:	My Monochromator Program.xrdml
<u>F</u> older:	C:W'Pert Data\My Name
<u>C</u> omment:	Example for Quick Start Guide
Sample	
ID:	Standard Si Sample
<u>N</u> ame:	Silicon Pellet
Prepared by:	My Full Name
Position	
<u>2</u> Theta (°):	28.000 _Phi (*): ∠(mm):
Offset (*):	0.000 Psi (*): Y (mm):
<u>O</u> mega (*):	14.000 ⊇ (mm):
Reflection	
Unit cell:	h <u>k</u> 1:
	OK Cancel Help

Press
 OK

The scan results are displayed as the measurement progresses.



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 pressing



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.

🚯 Change Sample S	tage	
Active <u>s</u> ample stage:	βpinner PW3064	
Change to sample stage:	Spinner PW3064	*
	Cancel	Apply

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



Remove the spinner and press

X'Pert Wizard		
∫Exchange sa	mple stage	
	Please mount the sample stage 'Capillary Spinner (manual)'.	~
	Previous	

- 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

🚯 New Progr	am	
<u>P</u> rogram type:	Absolute scan 🔽	ОК
		Cancel
		Help

- 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



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

🚯 Prepare Absolute Scan [Progra	m6]		
Configuration My Capillary Spinner Scan Axis 2Theta Other gonio angle Use actual omega at start Omega (*): 0000	Scan properties Repetition Step Continuous Pre-set counts Start angle (*): End angle (*): Step size (*): Time per step (s): Scan speed (*/s): Pre-set counts (counts): Number of steps: Total time (h:m:s):	5.000 75.000 0.020 0.50 0.040000 10000 3500 00:29:13	Comment Settings

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 (°2Theta):	2.122 (or the maximum
		allowed in your system)



🚯 Program 2: Settings		×
Filter: Actual Mask: Actual Mirror: Actual Monochromator: Actual		^
Soller slit: Actual	cted beam nath1	
PreFIX module: Actual	olog boam parm	
Anti-scatter siit: Actual	al	
Collimator: Actual	l	
Mode = Scanning	- ata) - 2 122°	
Filter: Actual	staj = 2.122	
Monochromator: Actua		×
X'Celerator[2]		~
A Celerator[2]		
Scan <u>m</u> ode:	Scanning 🔽	
Active length (*2Theta):	2.122	
OK Ca	ncel Apply He	lp)
3 OK .		

٠

Configuration Scan properties Repetition Comment My Capillary Spinner Step Settings Scan Axis © Continuous Settings 2Theta Pre-set counts Start angle (*): 5.000 Other gonio angle End angle (*): 75.000	🛣 Prepare Absolute Scan [Progra	m6]		
Omega (*): 0.000 Step size (*): 0.0167113 Time per step (s): 1.905 Scan speed (*/s): 1.114085 Pre-set counts (counts): 10000 Number of steps: 4189 Total time (h:m:s): 00:01:07	Configuration My Capillary Spinner Scan Axis 2Theta Other gonio angle O Use actual omega at start O Omega (*):	Scan properties Repetition Step Continuous Pre-set counts Start angle (°): End angle (°): Step size (°): Time per step (\$): Scan speed (°/s): Pre-set counts (counts): Number of steps: Total time (h:m:s):	5.000 75.000 0.0167113 1.905 1.114085 10000 4189 00:01:07	Comment Settings

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

5.4.3 Performing the Measurement

ť	Connect				×
ſ	Configuration				ОК
	Name	Description	Date	Owner	Cancel
	My Capillary Spinner	Configuration for Capillary Samples [USG]	21-Sep-2004	My Na	
	My MPD Spinner	Configuration for Powder Samples (USG)	21-Sep-2004	MyNa	Help
	My Other Optics	Example for Uther Analyses for Quick Start Guide	20-Sep-2004	MyNa	
	My Spinner My Triple Auia	Configuration for High Resolution for Quick Start Quide	21-Sep-2004	My Na My Na	
	My Thple Axis	Conliguiation for High Resolution for Quick Start Guide	20-5ep-2004	мума	
			1		
	<u> </u>			>	
l					

• 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:

X'Pert Data Collector	
 Incident beam path: Assuming radius: 240 mm Divergence slit. Assuming Prog. Div. Slit. Diffracted beam path: Assuming radius: 240 mm Diffracted Soller slit. Assuming Soller 0.02 rad Diffracted Filter. Assuming Nickel. Diffracted Anti-scatter slit. Assuming Prog. AS Slit. 	OK Cancel

• 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".



🚯 Instrument S	ettings	×
Position Sample	e Stage Sample Changer X-ray	
X-ray genera	tor	
Tension (kV):	45 Generator on	
<u>C</u> urrent (mA):	40	
X-ray tube		
∐-ray tube:	PW3373/10 Cu LFF DK137133 🛛 🗸	
	Breed	
Shutter		
	Line focus O Point focus	
	Shutter open	
	Lancel Apply	telp

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

🚯 Incident Beam Optics 🛛 🛛 🔀
Monochromator Beam Attenuator Filter Soller Slit Beam Knife PreFIX Module Divergence Slit Anti-scatter Slit Mask Mirror
Iype: Mirror Cu W/Si (parabolic MPD / Capillary Spinner) 💽
Default offset (*): -1.102
Lotal onset (): -1.115
OK Cancel Apply Help

• 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)".

X'Pert Data Collector	
▲ Please remove the Incident PreFIX module Progr. Div. Slit_Anti-scatter Slit and insert	OK
the Mirror Cu W/Si (parabolic MPD / Capillary Spinner).	Cancel

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".

🚯 Open Program				
List programs of type:	Absolute scan		~	ОК
Name	Description	Date	User	Cancel
My Capillary Program	Example for Quick Start Guide	21-Sep-2004	My Name	
My Program	Example for Quick Start Guide	21-Sep-2004	My Name	пер
<			>	

- 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.



🚯 Start		X
Program		
Name:	My Capillary Program	
Туре:	Absolute scan	
Description:	Example for Quick Start Guide	
File		
<u>N</u> ame:	My Capillary Measurement.xrdml	
<u>F</u> older:	C:WPert Data	
<u>C</u> omment:	Example for Quick Start Guide	
	×	
Sample		
<u>I</u> D:	Unknown Capillary	
<u>N</u> ame:	Capillary Sample	
Prepared by:	My Full Name	~
Position		
Diffractometer		
<u>2</u> Theta (*):	5.000 Ehi (*): ≚ (mm):	
Offse <u>t</u> (*):	41.513 P₂i (°): ⊻ (mm):	
<u>O</u> mega (°):	44.013 ≧ (mm):	
Reflection		
Unit cell:	h <u>k</u> 1:	
	OK Cancel Help	

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.

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).

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



	Close Control Form.
	Select what you want to do and press OK.
	Press Cancel to return to the Control Form.
	Close control window and go off-line
	OK Cancel
• Press	OK .

• Press 🛛 to leave X'Pert Data Collector.
4022 339 07601

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