

Using the Open Eularian Cradle (OEC) with the High-Speed Bragg-Brentano Optics on the PANalytical X'Pert Pro MPD

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This SOP and others are available at <http://prism.mit.edu/xray/education/downloads.html>

Remember, the X'Pert Pro MPD is a versatile instrument with many different optics and sample stages. The standard training for the X'Pert Pro MPD does not authorize you to change the PreFIX modules, which distinguish between the High-Speed configuration and the Parallel Beam configuration, or the Sample Stage. If you want to use a different configuration or sample stage, you must contact SEF staff to make the change for you.

NOTE: The PANalytical X'Pert PRO MPD has a new X'Celerator Scientific detector and the instrument control software has been upgraded from Data Collector v2.2 to v5.1. Changes are noted in ORANGE.

You are allowed to change the accessories for the PreFIX modules as instructed in this SOP.

- I. Configure the Instrument** **pg 3**
- II. Write a Measurement Program** **pg 9**
- III. Run the Measurement Program** **pg 11**
- IV. When You are Done** **pg 12**
- Appendix A. Terms and Conventions** **pg 12**
explains the conventions used for describing software items and actions.
- Appendix B. Instrument Safety** **pg 13-14**
provides detailed instructions on safety protocols for using this instrument.

PANalytical X'Pert Pro Operation Checklist

1. Engage the PANalytical_X'Pert in Coral	
2. Assess instrument status and safety	
a. Is the instrument on?	
b. Is the generator on?	Ch II
c. What is the tube power?	
d. Is the shutter open?	
3. Determine if the correct PreFIX optics and sample stage are on the instrument.	Ch V, Sect 3
a. If not, ask SEF staff to change them for you	
4. Start X'Pert Data Collector	Ch IV, Sect 2
5. Connect the computer to the instrument	Ch IV, Sect 2
6. Make sure the tube power is at stand-by level:	Ch V, Sect 2B
a. 40 kV and 10 mA for the Cu tube	
7. Choose and insert accessories (slits, filters, etc) into the PreFIX modules	Ch V, Sect 3
8. Load your sample	Ch V, Sect 3C
9. Specify the instrument configuration in Data Collector	Ch V, Sect 2D & E
10. Turn the tube power up to full power	Ch V, Sect 2B
a. 45 kV and 40 mA for the Cu tube	
11. Write a measurement program if you do not already have one	Ch VI, Sect 2
12. Run the measurement program	Ch VI, Sect 3
13. When finished	
a. Turn the tube power down to its stand-by level:	
i. 40 kV and 10 mA for the Cu tube	
b. Retrieve your sample	Ch VII
c. Clean the sample stage and sample holders	
d. Copy your data to a secure location	
e. Disengage the PANalytical in Coral	

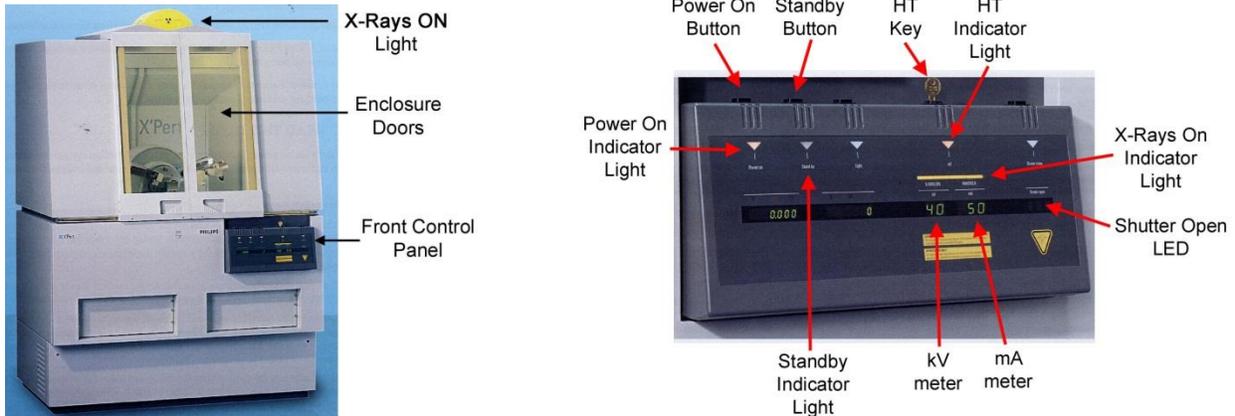
The Chapter number and section number on the right-hand side of the page indicate where you can find more detailed instructions in the X'Pert SOP. The notation used is Chapter Number in Roman numerals, Section in Arabic numerals, followed by subsections: for example, V.2.B refers you to Chapter V, Section 2, subsection B- which are the instructions for changing the tube power.

I. Configure the Instrument

This section walks you through the steps of setting up the instrument to begin your measurement. Many of these items are covered in more detail in the X'Pert SOP, sections IV and V.

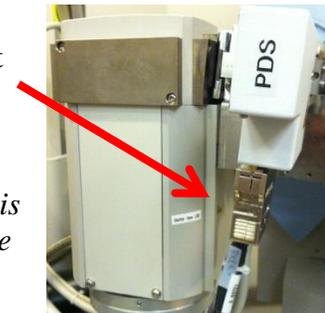
1. ENGAGE THE PANALYTICAL_XPERT IN CORAL

2. ASSESS INSTRUMENT STATUS AND SAFETY



- a. Determine if the generator is on.
 - i. The **X-rays ON** light should be lit- this light is found on the top of the instrument and on the front instrument panel (as shown above).
 - ii. If the generator is not on, contact SEF staff for help. We leave the generator on, even when the instrument is not in use—so if the generator is off then something is wrong.

- b. Determine if the shutter is open. If the shutter is open:
 - i. The yellow shutter open LED on the X-ray tube tower will be lit (shown to the right).
 - ii. The **Shutter Open LED** on the front panel (shown above) will display a number 1.
 - iii. *There is a triangle on the front panel labeled **Close Shutter**. This does not light up when the shutter is closed- it is just labeling the button that can be used to force the shutter to close if the computer crashes.*
 - iv. If the shutter is open, look at the Data Collector software to determine if a run is in progress.



1. If a measurement is in progress, either let it finish or Stop it
2. To stop a scan that is progress, click on the **Stop**  button in the Data Collector toolbar.



3. If a measurement is not in progress, something is wrong. Contact SEF staff for help.

3. DETERMINE IF THE CORRECT PREFIX OPTICS AND SAMPLE STAGE ARE ON THE INSTRUMENT

- a. Incident-beam PreFIX module should be the Prog. Divergence Slit (PDS)
- b. Diffracted-beam PreFIX module should be the X'Celerator attached to the Programmable Anti-Scatter Slit (PASS)
 - i. An optional monochromator is available for the X'Celerator if you are working with samples that contain Fe or Co; or if you are doing slow scans for Rietveld refinement. Talk to SEF staff if you are interested in using this. The monochromator will replace the PASS PreFIX module.
- c. The sample stage should be the OEC.
- d. *If the correct modules are not installed, ask SEF staff to change them for you*

4. READY THE INSTRUMENT

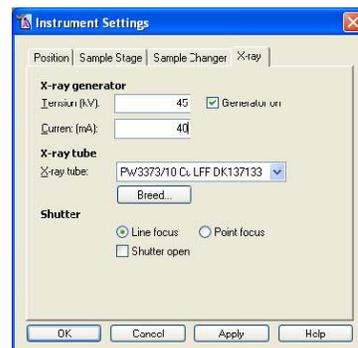
- a. If the program X'Pert Data Collector is already running, quit it
- b. Start the program **X'Pert Data Collector**
 - i. Enter your user name and password to log in to your account
 1. Username is the same as your Kerberos account, password is **soap**
 - ii. Select menu *Instrument*>*Connect* to connect the computer to the instrument
 - iii. Select the configuration "**OEC Motorized Platform**" and click **OK**
 - iv. Click **OK** in the next window that opens, which is a status message that tells you what PreFIX modules the software thinks are currently installed.
 1. *You may see an error message if the person before you changed the sample stage but did not update the data collection software. If this happens:*
 - a. go to the menu *Tools*>*Exchange Sample Stage*
 - b. In the dropdown menu *Change to sample stage*, select the option "**Open Eularian Cradle (manual Z)**" and click **OK**
 - c. Click **Next** in the next several dialogue boxes, then click **Finish** in the last dialog box.
 - d. It may take a few minutes for the stage to initialize, but when done the dialog window will close. Then repeat step iii above.

- c. Make sure the tube power is at **40 kV** and **10 mA**

- i. The Generator Power is shown in the Instrument Settings tab

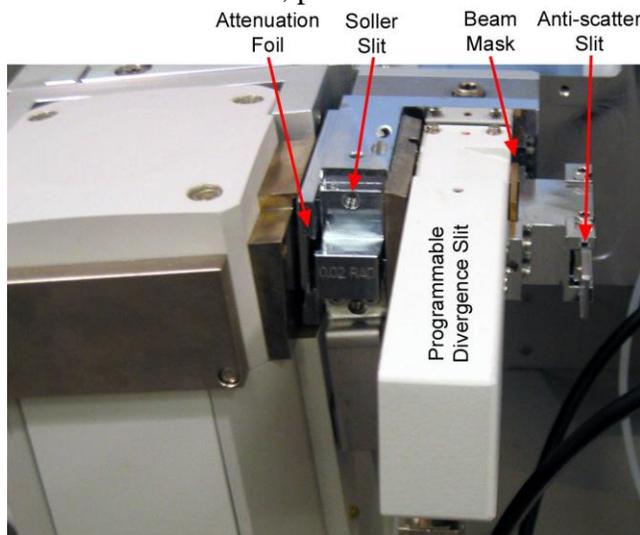


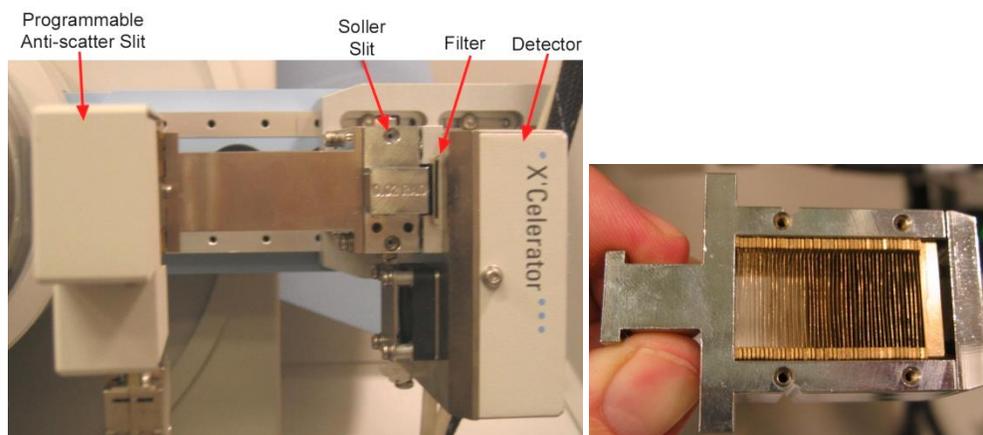
- ii. To change the power, double-click on the item **Generator:MPPC** to open the *Instrument Settings* window
- iii. In the X-ray tab, change the **Tension (kV)** and **Current (mA)** to the desired values, then click **OK**.
 1. The Tension should be 40 kV
 2. The Current should be 10 mA



5. INSERT THE APPROPRIATE OPTICS AND ACCESSORIES FOR THE PREFIX OPTICS

- a. The incident-beam side optic should be the Programmable Divergence Slit (PDS)
 - i. Decide if you will use the PDS in fixed or automatic mode
 1. Use the spreadsheet *PDS Guidance Calculator.xlsx* to plan your PDS choice
 2. Fixed mode is usually used for infinitely thick samples (thicker than 100 microns). Typical fixed slit sizes are $\frac{1}{4}$ or $\frac{1}{2}$ deg.
 3. Automatic mode is usually used for thin samples (thinner than 10 microns). Typical illuminated lengths are 8 to 4mm.
 - ii. Insert the Incident-Beam Anti-Scatter Slit that matches the PDS.
 1. The *PDS Guidance Calculator* will tell you the correct anti-scatter slit size.
 2. Hold the antiscatter slit by the numbered end, with the numbers upside down.
 3. As you push the anti-scatter slit in, you need to lift up slightly on the end that you are holding. Push until the slit clicks into place.
 - iii. Select and insert a Soller Slit
 1. A larger Soller slit gives more intensity, while a smaller Soller slit produces better peak shapes at lower angles 2θ
 - a. The 0.04rad Soller slit is most commonly used
 - b. The 0.02 or 0.01rad Soller slits are used for Reitveld quality data or for samples with diffraction peaks below $20\text{deg } 2\theta$
 2. You must insert the Soller slit with a shutter lever (i.e. leg)
 3. Hold the Soller slit by the handle and push it straight in.
 - iv. Select and insert a width limiting Beam Mask for your sample
 1. The 10mm mask is most commonly used; other sizes are 5, 15, and 20mm.
 2. To remove the Beam Mask, you need to feed a small torque wrench through the hole at the top center of the mask and then pull it out using the wrench.
 3. To insert the Beam Mask, push down until it clicks into place





b. Diffracted-beam side should use the X'Celerator detector

- i. Insert the Soller slit that matches the incident-beam side
 - 1. Use the Soller slit that does not have facing plates, pictured above.
- ii. Insert the appropriate Beta Filter
 - 1. use the Ni Filter for Cu radiation
 - a. an optional monochromator is available. This may be recommended if your sample is mostly Fe or Co or if your data are intended for Rietveld refinement
 - b. the monochromator must be inserted by trained SEF staff

c. Load the sample into the instrument

- i. Turn the digital height gauge on by pressing the green button.
- ii. Attach the digital height gauge to the OEC and tighten the knurled knob
 - 1. There is an alignment slot in the base of the height gauge that slides over the alignment pin on the OEC post. The gauge probe will be centered on the OEC.
- iii. Position your sample under the height gauge
 - 1. If your sample is a powder in a holder, put the gauge on the edge of the holder when adjusting the height rather than on the powder.
 - 2. If your sample is delicate, you can adjust the height of the stage itself to 0mm minus the thickness of your sample; or use an equivalent sample to adjust the height (such as a substrate with no film).
 - 3. If your sample is small (less than 15mm x 15mm), consider putting it on a glass slide to prevent artifact peaks from the OEC sample stage.
- iv. Rotate the OEC stage to adjust the sample height until the gauge reads 0.00mm
- v. Loosen the knurled knob that secures the height gauge to the OEC stage.
- vi. Center your sample underneath the gauge—this is where the X-ray beam is centered.
 - 1. If your sample is powder in a holder, or has a delicate surface:
 - a. Use the lever to raise the gauge probe so that it is not touching the sample.
 - b. Center the powder underneath the probe.
 - c. While continuing to use the lever to raise the probe off of the sample, remove the gauge from the OEC.
- vii. Turn the gauge off by holding in the green button for a few seconds.



6. CHANGE THE INSTRUMENT CONFIGURATION IN X'PERT DATA COLLECTOR

Remember to click **Apply** every time you make a change in the Optics configuration

- a. Select the Incident Beam Optics tab in the *Instrument Window* (on the left-hand side of Data Collector)

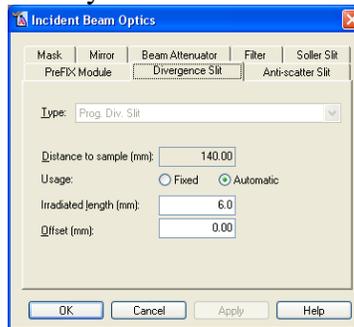
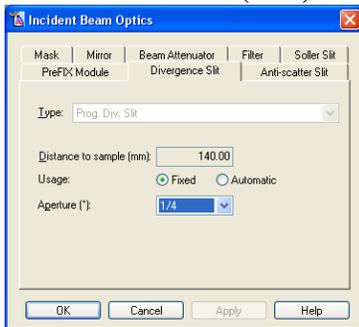


- i. In the *Instrument Window*, double-click on the item **Incident Beam Path** to open the *Incident Beam Optics* window

- ii. In the PreFIX Module tab, set **Type** to “**Prog. Div. Slit & Anti-scatter Slit**”

- iii. In the Divergence Slit tab, select the Fixed or Automatic mode for the computer-controlled divergence slit.

1. Usage “**Fixed**” will maintain a constant divergence angle, which is selected in the **Aperture** (°) drop-down menu
2. Usage “**Automatic**” will maintain a constant irradiated length, which can be entered into the **Irradiated length (mm)** field
 - a. **Offset (mm)** should always be **0**



Remember, typical Fixed slits sizes are 1/2 to 1/4 deg, and typical Automatic slit lengths are 4 to 8mm.

- iv. In the Anti-scatter Slit tab, use the **Type** drop-down menu to indicate what size Anti-scatter slit you inserted into the instrument.

- v. In the Mask tab, use the **Type** drop-down menu to indicate what size mask you inserted into the instrument.

- vi. In the Mirror tab, set the **Type** to “**None**”

- vii. In the Beam Attenuator tab, set the **Type** to “**None**”

- viii. In the Filter tab, set the **Type** to “**None**”

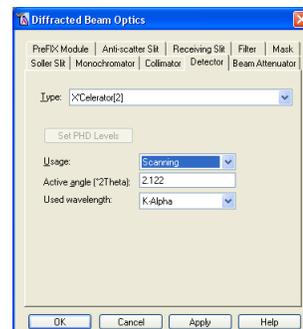
- ix. In the Soller Slit tab, use the **Type** drop-down menu to indicate what size Soller slit you inserted into the instrument.

- x. Click **OK** to close the dialog window

b. Configure the Diffracted Beam Optics tab in the *Instrument Window*

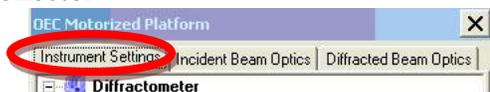


- i. Double-click on the item **Diffracted Beam Path** to open the *Incident Beam Optics* window
- ii. In the PreFIX Module tab, set the **Type** to “**X’Celerator**”
 1. if using the monochromator, set the **Type** to “**X’Celerator & Monochromator Cu**”
- iii. In the Anti-scatter Slit tab, set to match *the incident-beam divergence slit*
 1. Select “**Prog AS Slit**” from the drop-down menu for **Type**
 2. With **Usage** “**Fixed**” the **Aperature** (°) should be identical to that for the incident-beam Programmable Divergence Slit
 3. With **Usage** “**Automatic**” the **Observed Length (mm)** will be the same as the **Irradiated length (mm)** for the incident-beam Divergence Slit
- iv. In the Receiving Slit tab, set the **Type** to “**None**”
- v. In the Filter tab, set the **Type** to “**Nickel**”
- vi. In the Mask tab, set the **Type** to “**None**”
- vii. In the Soller Slit tab, use the **Type** drop-down menu to indicate what size Soller slit you inserted into the instrument.
- viii. In the Monochromator tab, set the **Type** to “**None**”
 1. The Monochromator **Type** will be set to “**Diffr. Beam Flat 1x graphite for Cu (X’Cel)**” if using the optional monochromator.
- ix. In the Collimator tab, set the **Type** to “**None**”
- x. In the Detector tab
 1. Set **Type** to “**X’Celerator[2]**”
 2. Set **Usage** to “**Scanning**”
 3. Set **Active angle (°2Theta)** to “**2.122**”
 4. Set **Used Wavelength** to “**K-Alpha**”
- xi. In the Beam Attenuator tab, set the **Type** to “**None**”
- xii. Click **OK** to close the dialog window

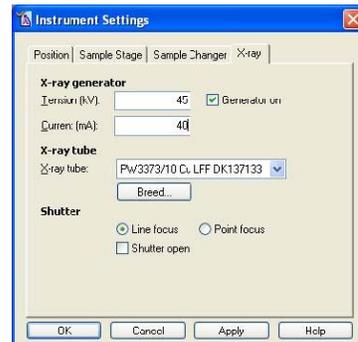


7. TURN UP THE GENERATOR POWER TO **45 kV** AND **40 mA**

- a. Select the tab Instrument Settings tab, found in the Instrument Window on the left-side pane in X'Pert Data Collector



- b. To change the power, double-click on the item **Generator:MPPC** to open the *Instrument Settings* window
- c. In the X-ray tab, change the **Tension (kV)** to **45** and **Current (mA)** to **40**, then click **OK**



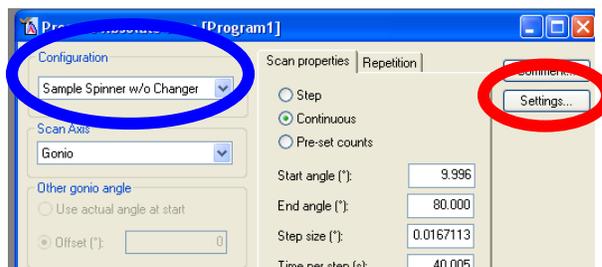
II. Write a Measurement Program

- If you already have a suitable measurement program written, you can skip this step and go to Section III, “Run the Measurement Program”, on page 11.
- Most data are collected using an Absolute Scan, as described below. Other types of programs are described in the X'Pert SOP.
- You can open a previously saved program and modify it, instead of starting from scratch. Go to *File>Open* to open the program; save by going to *File>Save As...*

WRITING AN ABSOLUTE SCAN PROGRAM

- a. Select menu *File> New Program*
- b. Choose “**Absolute Scan**” from the **List programs of type:** drop-down menu and then click **OK**
- c. In the *Prepare Absolute Scan* window, specify the **Configuration** as “**OEC Motorized Platform**” using the drop-down menu (circled in blue to the right)

- d. Click on the  button (circled in red).
- The **Settings** button is in the upper right corner of the *Prepare Absolute Scan* window.
 - The Program Settings window will open (shown on the next page)

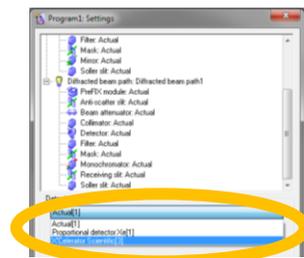


This step is important!! If you forget this step when using the X'Celerator high-speed detector, your data will not be collected properly.

iii. in the **Program Settings** window, scroll down until you can select **Detector** in the Diffracted beam path

iv. Once you select **Detector**, an information field will appear in the bottom of the **Program Settings** window

v. In the drop-down menu, shown below, change the value for **Detector** from “**Actual**” to “**X’Celerator Scientific[3]**”



vi. Specify for parameters for the X’Celerator Detector

1. Set **Scan mode** to “**Scanning**”
2. Set **Active Length** ($^{\circ}2\theta$) to “**2.122**”
 - a. Use a smaller active length, such as “**0.518**”, when collecting data below $3^{\circ}2\theta$. This reduces background noise, but will make the scan take longer.

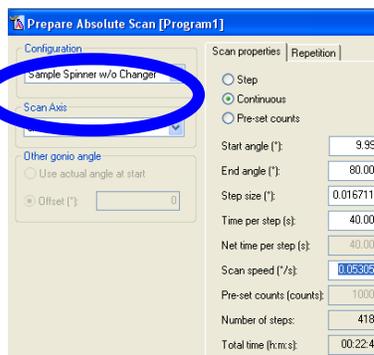
vii. Click **OK**

e. In the **Prepare Absolute Scan** window, specify the **Scan Axis** (circled in blue below)

i. “**Gonio**” scans are the conventional Bragg-Brentano parafocusing geometry, where ω is always $\frac{1}{2} 2\theta$. This is the most common Axis.

ii. “ **2θ - ω** ” is used when collecting data from a film on a single crystal substrate.

1. 2θ and ω are varied so that $\omega = \frac{1}{2} 2\theta + \text{offset}$.
2. Set **Offset**($^{\circ}$) to any nonzero value between 5 and -5 to reduce or eliminate the strong intensity and artifact peaks from the substrate.
 - a. An **Offset**($^{\circ}$) value of “**1**” is the most common.



f. Set the **Scan Properties**

i. The Scan Mode is always “**Continuous**”

ii. Specify the **Start angle**($^{\circ}$), **End angle**($^{\circ}$), and **Step size**($^{\circ}$)

1. The typical **Step size** is “**0.0167113**”.
2. For the **Start angle**, **End angle**, and **Step size**, the actual value used may differ slightly from what you enter- depending on physical constraints of the detector

iii. Specify the scan rate

1. You can either enter a value for **Time per step** (s) or **Scan speed** ($^{\circ}$ /s). The other value and the **Total time** will be recalculated accordingly.
2. Typical **Time per step** (s) values are:
 - a. **5 to 20 sec/step** for a fast scan of a well crystallized material or for a preliminary scan
 - b. **20 to 80 sec/step** for a detailed scan of a well crystallized material
 - c. **80 to 200 sec/step** for a slow scan of a complex sample, a poorly crystallized sample, or a nanocrystalline thin film
 - d. **200 to 1000 sec/step** for a very slow, detailed scan of a very complex sample with the data intended for very demanding analysis

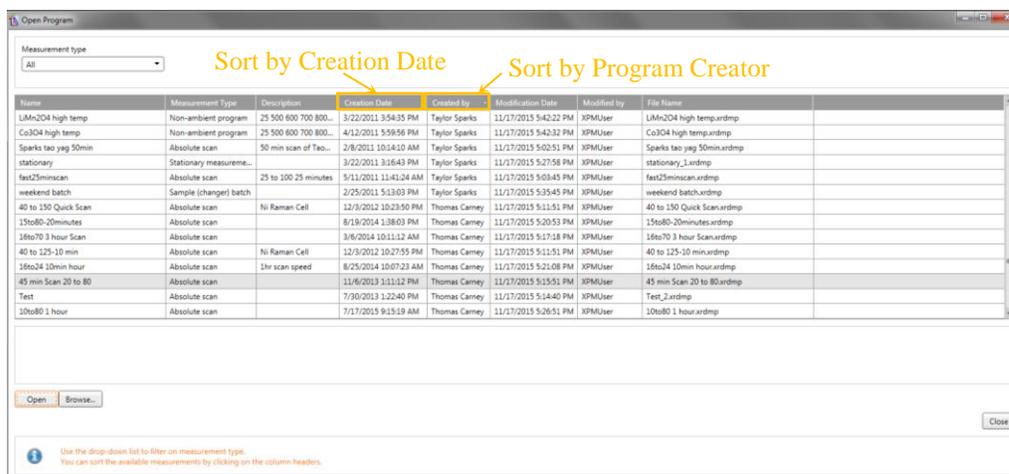
- i. With a very long scan, you might get better data using the diffracted-beam monochromator instead of the beta-filter. This will avoid anomalies in the background due to the beta filter absorption edge.
 - ii. Instead of collecting a single slow scan, consider collecting several faster scans. For example, instead of one 8-hour scan collect four 2-hour scans. You can set a scan to repeat in the Repetition tab of the *Prepare Absolute Scan* window.
- g. Save the measurement program
- i. Go to *File > Save*
 - ii. Write a **name** and **description** for the measurement program
 1. the same program can be used for many different samples, so it is recommended that you not use the name of a specific sample
 2. I generally designate the scan parameters in the program name and the general use of the program in the description, for example:
 - a. **Name:** “High-Speed 20to80deg in 30min”
 - b. **Description:** “Detailed scan of normal oxide powder”
 3. Click **OK** to save the program.

III. Run the Measurement Program

1. In the main menu, select *Measure > Program...*
2. In the *Open Program* window,
 - a. Select “**Absolute Scan**” in the **List programs of type:** drop-down menu
 - b. Select the measurement program that you want to use and click **OK**

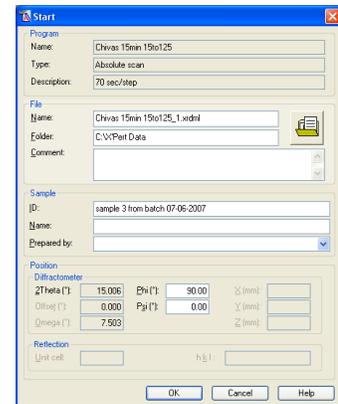


NOTE: All previous measurement programs are in the C:\PANalytical\Data Collector\Programs



3. The *Start* window will open
4. Specify the **File Name, Folder, and Sample ID**
 - a. You can create a personal folder in **C:\XRD Data**

- i. The **C:\XRD Data** folder is shared across the network
 - ii. Click on the folder icon to graphically navigate to the folder where you want your data saved
 - b. **Sample ID** will show up as a description header in most data analysis programs
 - c. **Comment, Name,** and **Prepared by** are rarely useful to complete
5. Click **OK**
- a. The measurement will start. The doors will lock and the shutter will open.
 - b. If the doors do not lock properly, you will get an error message. Make sure the doors are completely closed, then try starting the scan again.
 - c. Data will not shown in realtime on the screen until a few hundred data points have been collected. Therefore, you might not see a plot until a few minutes after you start the scan.
 - d. Data are automatically saved every 5 minutes and at the end of the scan.
 - e. When the scan finishes, the shutter will close and the doors will unlock.



IV. When You are Done

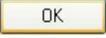
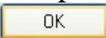
1. When the measurement finishes:
 - a. Your data are automatically saved
 - b. The shutter is automatically closed
2. Turn down the generator power to **40 kV** and **10 mA**
3. Remove your sample
4. Quit X'Pert Data Collector
5. Disengage the PANalytical_XPert in Coral

Appendix A. Terms and Conventions Used

In this section, we describe the terms and conventions used in this SOP and how they relate to the user interface of the Data Collector software.

Terms Used to Denote an Action

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

Click	Press the left mouse button and quickly release it.
Double-click	Press the mouse button twice (quickly) on an icon, item, file, etc.
Right-click	Press the right mouse button and quickly release it.
Check/Uncheck	Click in a check box (<input checked="" type="checkbox"/>) to check it or uncheck it
Enter	Type in information. This can be either text or numerical data.
Press	Press a key on the keyboard, or a push-button in a window.
Select	Move the mouse cursor to the option you want and click the left mouse button.
Toggle	Switch between parameters or states (for example: On-Off-On).
Press 	In the examples in this Guide we terminate most actions by saying “press  ”; you can usually press the Enter key instead.

click (or press) 	The instruction to click (or press)  is used in this Guide as an instruction to close the window that you are currently working in, not the program. The instructions may instead say “close this window”
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Program Parameters

- Menu items are printed in *italics*, for example: *File, Edit*, etc
- Nested menus and selections within menus are indicated by >, for example *File>Open*, *Control>Preferences*
- Window titles (shown in the blue title bar of each window) are written in ***bold italics***.
- Names of tabs are underlined.
- Icons or Buttons on a dialogue box that you should click are written in **bold** text or the actual button graphic is shown (for example: **Apply** or )
- All fields are shown in bold between “**quotation marks**”.

Appendix B. Instrument Safety

Pictured below, in Figure 2.1, is the X'Pert Pro MPD. There are three primary features that you should be aware of. The **X-Rays On** light on top of the diffractometer is your first safety indicator; it is lit when the generator is turned on. The front control panel includes additional safety and instrument status indicators. A close-up of the front control panel is shown in Figure 2.2. The enclosure doors are leaded so that they are radiation shielding. Before the shutter can open, these doors will lock—thereby preventing you from entering the enclosure when there is a danger of exposure.

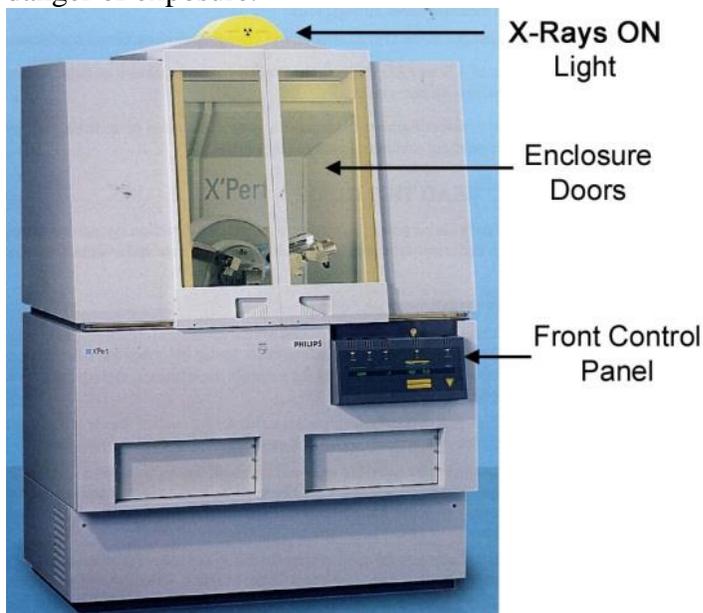


Figure 2.1 The enclosure for the X'Pert Pro MPD

There are four safety questions to ask yourself when using the PANalytical X'Pert Pro MPD. They are: “is the instrument on?”, “is the generator on?”, “what is the tube power?”, and “is the shutter open?”

Is the instrument on?

When the instrument is off, either the **Standby** indicator light on the front control panel (Fig 2.2) will be lit or no lights on the front control panel will be lit.

When the instrument is on, the **Power On** indicator light will be lit.

When the instrument is on, the generator is not necessarily on. If the generator is not on, no X-rays will be produced and the instrument is safe.

Is the generator on?

When the generator is on, the large **X-Rays On** light on top of the diffractometer will be lit (Fig 2.1). The **X-RAYS ON** indicator light on the front control panel will also be lit.

Even if the generator is on no X-rays will be produced if the tube power is low.

What is the tube power?

The tube power is indicated on the front control panel by the kV and mA digital meters. The tube power may also be indicated by the X'Pert Data Collector software if it is running.

The lowest power is 15 kV and 10 mA. When at this power, few X-rays are produced by the X-ray tube. Stand-by power is 45 kV and 20 mA. At this setting, a low power X-ray beam is produced. This is the power you should set the generator to when you are finished with your data collection. The operating power is 45 kV and 40 mA for the Cu tube and 40 kV and 40 mA for the Co tube. At this power, the X-ray tube is producing the maximum flux of X-rays. The instrument is still safe as long as the shutter is closed.

Is the shutter open?

When the shutter is open, the yellow-green shutter open light on the X-ray tube tower will be lit. Also, the **Shutter Open** LED on the front panel will display a vertical line. Most importantly, when the shutter is open the doors to the diffractometer enclosure will be locked. This means that you cannot get into the instrument while the shutter is open. You should still look at the shutter open light before attempting to open the doors, however, just to be cautious.

When the shutter is closed, no X-rays are leaving the X-ray tube tower and the instrument is safe. However, when working inside the instrument (to change samples, to change accessories) you should lower the tube power to 40 kV and 10 mA in order to lessen the chance of radiation exposure.

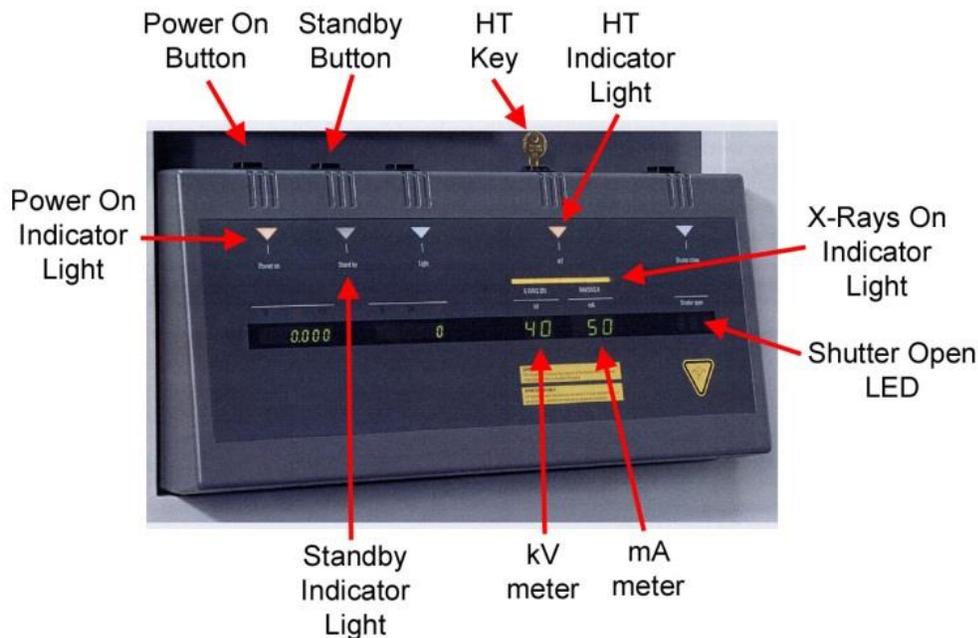


Figure 2.2 The front control panel for the X'Pert Pro MPD.