PANALYTICAL EMPYREAN

Powder X-Ray Diffractometer

#	Revised By	Date	Modification
1	Katherine M. Marczenko	October 8 2021	Initial Release

This document was adapted from a Standard Operating Procedure available through the Materials Research Laboratory at Massachusetts Institute of Technology.

1   Introduction

The following is a guide for collecting data using the Panalytical Empyrean Powder X-Ray diffractometer located within the Department of Chemistry at the University of Guelph. This document is intended as a "walk-through" user guide geared towards novice users. A basic knowledge of the fundamentals of diffraction and crystallography is expected.
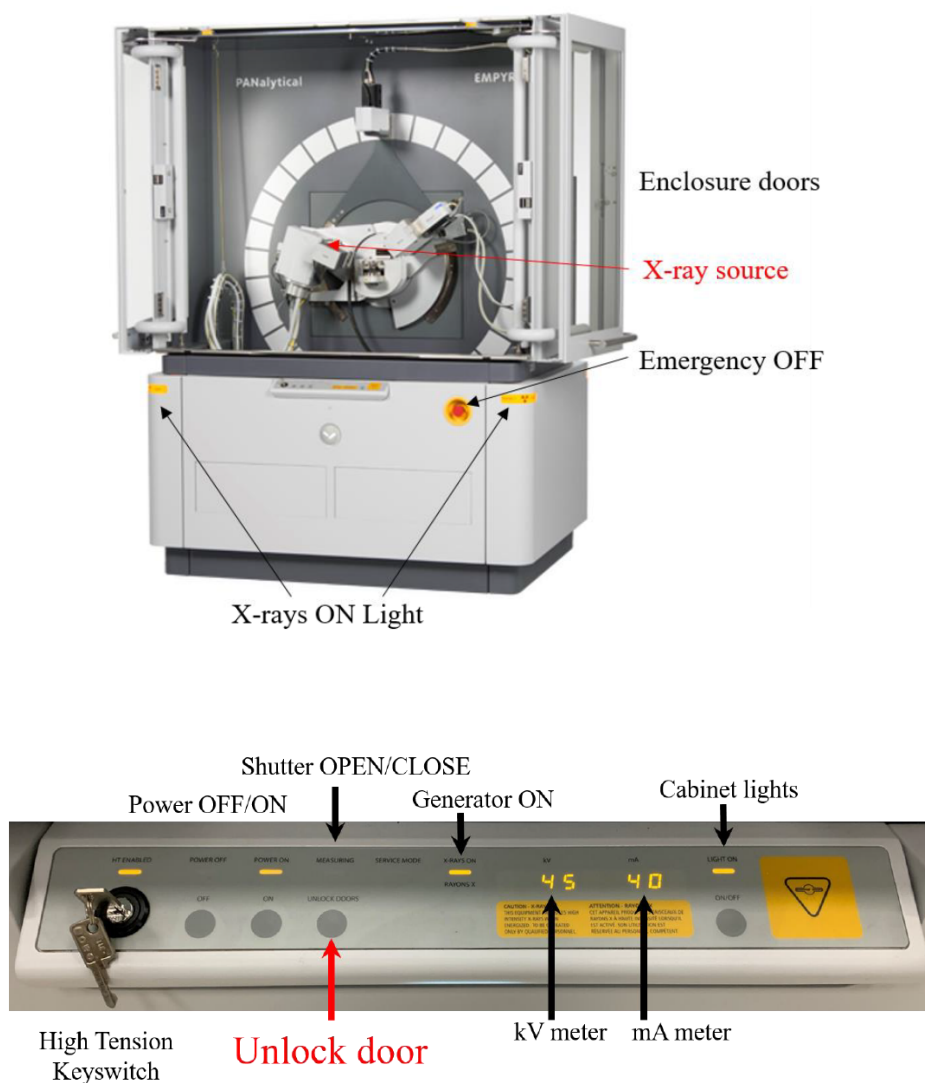
2   Configuring the Instrument

Assess Instrument Status and Safety.

The X-ray generator is on when the X-ray light is lit. This light is found on the corners of the instrument and on the front instrument panel, as shown in the above image. If the generator is not on, contact Facility Staff for help. The generator is left on, even when the instrument is not in use. If the generator is off, then something is wrong.


The X-ray shutter is open when the yellow shutter open LED on the X-ray tube tower is lit (image to the left). The **Measuring** indicator light on the front panel will display a number 1. If the shutter is open, look at the Data Collector software to determine if a run is in progress.

If a run is in progress, check the scheduling system (Skedda) and the PXRD log book to determine if there are scheduling conflicts. If there are, please contact the Facility Manager. Do NOT stop a scan that is in progress without explicit permission from the Researcher who began the experiment.

If a measurement is not in progress, but the shutter is open, something is wrong. Contact the Facility Manager for help.
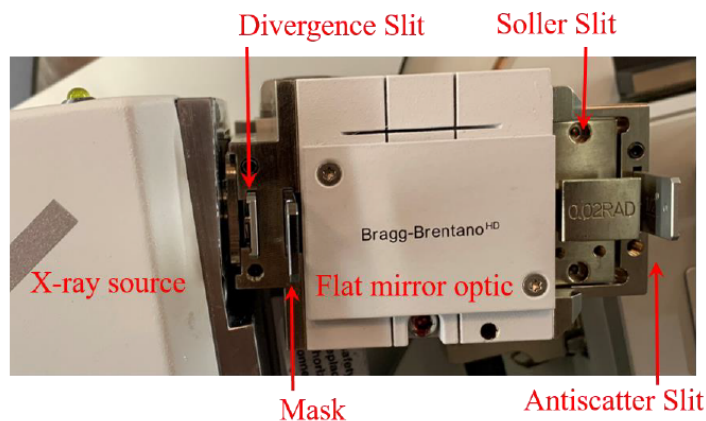
Ready the Instrument.

1. Start the program **Data Collector**. Enter the username (user-1) and password (galaxy) to log in.

2. Select menu ***Instrument > Connect*** to connect the computer to the instrument.

3. Select the configuration "**Reflection-transmission spinner**" and click **OK**.
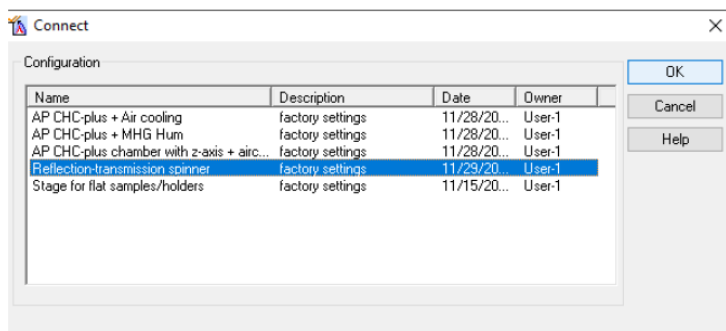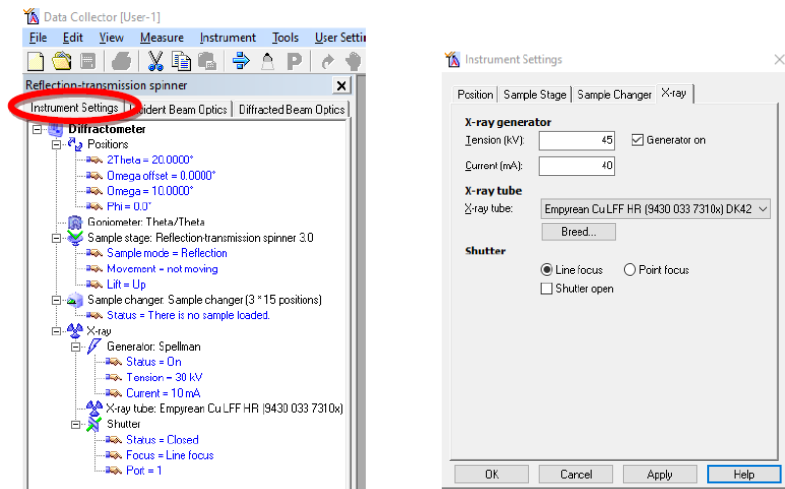
4. Click **OK** in the next window that opens, which is a status message that tells you what PreFIX modules the software thinks are currently installed. If you need to change any of the modules, please contact the Facility Manager.

5. Make sure the tube power is at **30 kV** and **10 mA**. The Generator Power is shown in the Instrument Settings tab. To change the power, double-click on the item **Generator:Spellman** to open the *Instrument Settings* window.

6. In the X-ray tab, change the **Tension (kV) and Current (mA) to the desired values, then click OK.** The ramp rate is located on the title page of this document.

The below image shows the optics accessories available for the instrument. If you would like to change any of these accessories, please contact the Facility Manager.

3   Data Collection

You can now load the sample into the instrument. ***Make sure samples are flush to the top of the sample holder.*** Failure to do so may spill the sample in the instrument or damage the accepting reflection-transmission spinner. A tray to catch any spilt sample is located on the floor of the instrument case. Please ensure that it is clean after each use.

Write a Measurement Program.

If you already have a suitable measurement program written, you can skip this step and go to "*Run the Measurement Program*". Most data are collected using an Absolute Scan. 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...*

To write a new absolute scan program, follow the below steps:

1. Select menu *File > New Program*

2. Choose "**Absolute Scan**" from the **List programs of type:** drop down menu and then click **OK**

3. In the ***Prepare Absolute Scan*** window, specify the **Configuration** as "**Reflection-transmission Spinner**" using the drop-down menu Click on the "**Settings**" button in the upper right corner of the ***Prepare Absolute Scan*** window.
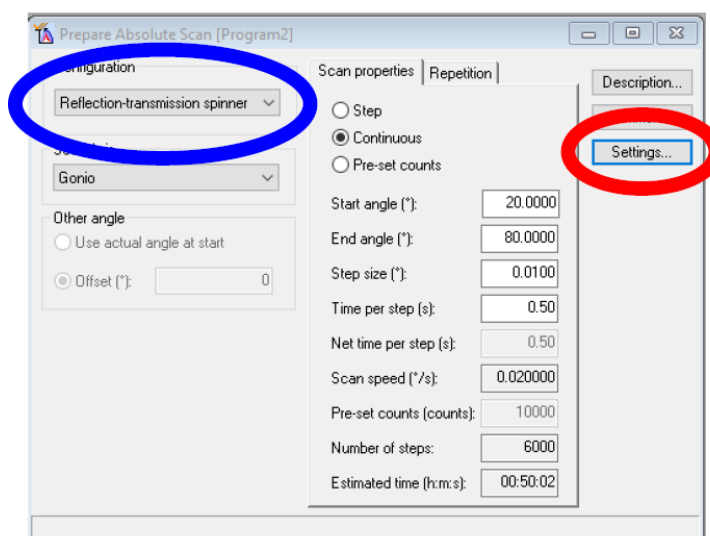
    (a) In the ***Program Settings*** window, select "**Movement = not moving**" in the Sample Stage: Reflection-transmission Spinner 3.0 menu. Click radio button: "Spinning, Revolution time = **16s**".

(b) In the ***Program Settings*** window, scroll down until you can select Detector in the Diffracted beam path

(c) Once you select Detector, an information field will appear in the bottom of the Program Settings window

(d) In the drop-down menu, shown below, change the value for Detector from "**Actual**" to "**PIXcel1D-Medipix3 detector[1]**"

(e) Specify for parameters for the X'Celerator Detector by setting Detector mode to "*Scanning line detector (1D)*", *Active Length* (°2Theta) to "3.3482", and clicking **OK**.

4. In the Prepare Absolute Scan window, specify the Scan Axis. "**Gonio**" scans are the conventional Bragg-Brentano parafocusing geometry, where omega is always $\frac{1}{2}$ 2Theta. This is the most common Axis."**2Theta-Omega**" is used when collecting data from a film on a single crystal substrate.

   (a) With this instrument, 2Theta and Omega are varied so that Omega = $\frac{1}{2}$ 2Theta + offset.

   (b) Set Offset(°) to any nonzero value between 5 and -5 to reduce or eliminate the strong intensity and artifact peaks from the substrate. An Offset(°) value of "1" is the most common.

5. Set the **Scan Properties**.

   (a) The Scan Mode is always "**Continuous**".

   (b) Specify the Start angle(°), End angle(°), and Step size(°). The typical Step size is "0.0131303".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.

   (c) Specify the scan rate. You can either enter a value for Time per step (s) or Scan speed (°/s). The other value and the Total time will be recalculated accordingly.Typical Time per step (s) values are 5 to 20 sec/step for a fast scan of a well crystallized material or for a preliminary scan; 20 to 80 sec/step for a detailed scan of a well crystallized material; 80 to 200 sec/step for a slow scan of a complex sample, a poorly crystallized sample, or a nanocrystalline thin film;200 to 1000 sec/step for a very slow, detailed scan of a very complex sample with the data intended for very demanding analysis.

6. Save the measurement program by navigating to *File > Save*. Write a **name** and **description** for the measurement program. The same program can be used for many different samples, so it is recommended that you not use the name of a specific samples. We recommend designating 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"

Run the Measurement Program.

You can now open and run the measurement program you have created. In the main menu, select *Measure > Program*. The ***Open Program*** window will appear and allow you to select "**Absolute Scan**" in the **List of programs of type:** drop-down menu. Select the program you want to use and click OK.

The **Start** window will open. Specify the **File Name**, **Folder**, and **Sample ID**. Click **OK**. *This will start the measurement*!

The doors will lock and the shutter will open. 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. Data will not show 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. When the scan finished, the shutter will close and the doors will unlock.
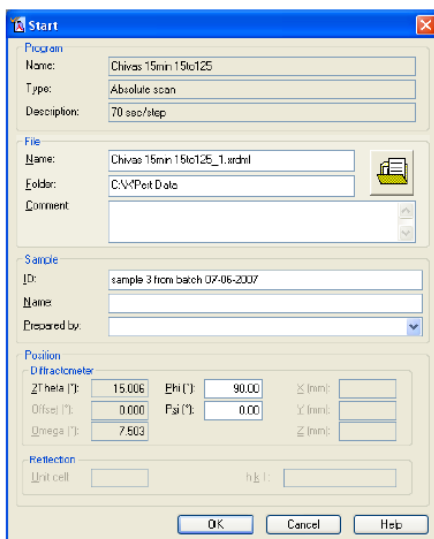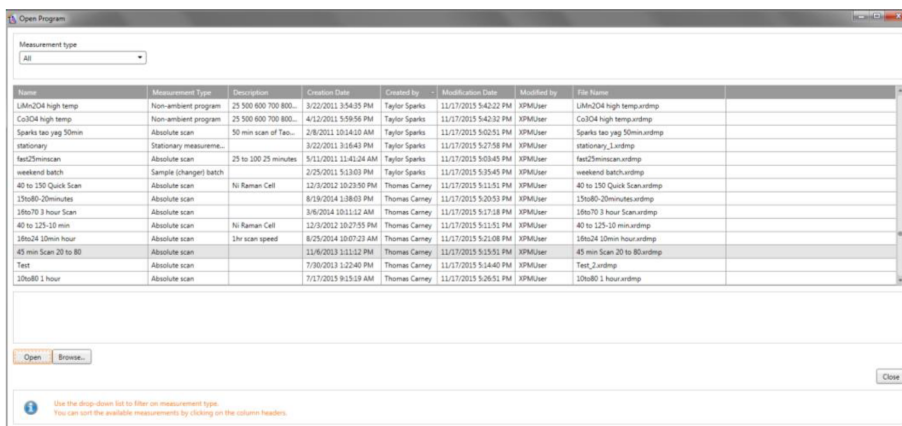
4   Shutting Down

When the measurement finishes, your data is automatically saved and the shutter is automatically closed. Turn down the generator power to 30kV and 10mA using the same ramping procedure as before. Do NOT turn of the X-ray genertor or the instrument. Remove your sample from the diffractometer and quit Data Collector.