SIP | Analytical & Diagnostics Lab

PANALYTICAL X'PERT PRO: Quick Start Guide

The X'Pert Pro is a versatile X-ray Diffraction (XRD) system with interchangeable "PreFIX" modules. The default configuration enables basic measurements, such as θ-2θ scans for phase identification. The standard training for the X'Pert Pro does not authorize you to change the PreFIX modules or the Sample Stage. If you require a different configuration, contact ADL staff to make the change for you.

1. Starting up the system

1. Log onto the instrument PC.

2. Open the **Data Collector** software.

3. On the top toolbar, select "**Instrument**", then select "**Connect**" in the drop-down.

   a. On the pop-up, select the "**USER**" configuration and "**Triple Axis / Coll. 0.18**" radio button, then click **OK**.

   b. Another pop-up will alert you to the assumed instrument settings. Click **OK**.

4. Once connected, check the toolbar that has appeared on the left.

   a. On the "**Incident Beam Optics**" tab, set the <u>Beam attenuator</u> usage to "**Do not switch**" and select the "**Activated**" checkbox (if it is not already).

   b. On the "**Instrument Settings**" tab, set the <u>Generator</u> to **45 kV / 40 mA**.

   If the XRD hasn't been used for more than 24 hours, you may want to run the "fast" ($\sim$6 min) X-ray gun breeding procedure via the button in the "X-ray Settings" pop-up.

2. Mounting samples

<u>Solid samples</u> can be mounted using double-sided tape or, if they are light and thin enough, the three sliding clips on the basic sample stage.

<u>Powder samples</u> must be consolidated in some way; either by spreading a uniform layer onto double-sided tape, pressing into a solid pellet using a pellet press, etc.

You may want to mount your sample on an amorphous material, such as a glass slide, to reduce potential spectral contamination from the aluminum mounting surface.

3. Aligning samples

1. Ensure the Beam attenuator usage is set to "**Do not switch**" and "**Activated**".

2. Ensure the sample stage is at its home position (all stage positions set to zero).

3. Ensure the beam optics are appropriate for alignment.
   a. Insert the appropriately sized **vertical beam mask** into the incident beam optics.
   b. Insert the **1/32° divergence slit** into the incident beam optics.
   c. Insert the **0.18° parallel plate collimator slit** into the diffracted beam optics.

4. Begin by aligning 2θ.
   a. On the top toolbar, select "**Measure**", then select "**Manual Scan**" in the drop-down.
      i. On the pop-up, select "**2Theta**" as the Scan Axis.
      ii. Type in a Range of **1°**, Step size of **0.01°**, and Time per step of **0.2 s**.
      iii. Then select "**Start**" and wait for the scan to finish.
   b. Right click on the completed scan and select "**Move Mode**" from the context menu.
   c. Click and drag the **vertical green line** onto the peak maximum.
   d. Close the scan.

5. Now, manually move the **Z** position to approximately the correct position for your sample thickness (typically somewhere between 6 - 10 mm).

6. Next, align **Z**.
   a. On the top toolbar, select "**Measure**", then select "**Manual Scan**" in the drop-down.
      i. On the pop-up, select "**Z**" as the Scan Axis.
      ii. Type in an appropriate Range, Step size, and Time per step. Try to pick values that keep the total scan time under 1 minute.
      iii. Then select "**Start**" and wait for the scan to finish.
   b. Right click on the completed scan and select "**Move Mode**" from the context menu.
   c. Click and drag the **vertical green line** onto the midpoint of the intensity dropoff.
   d. Close the scan.

7. Finally, align Ω.

    a. On the top toolbar, select "**Measure**", then select "**Manual Scan**" in the drop-down.

        i. On the pop-up, select "**Omega**" as the Scan Axis.

        ii. Type in an appropriate Range, Step size, and Time per step. Again, try to keep the total scan time under 1 minute. Typically you will need a Range greater than **3 - 4°**.

        iii. Then select "**Start**" and wait for the scan to finish.

    b. Right click on the completed scan and select "**Move Mode**" from the context menu.

    c. Click and drag the **vertical green line** onto the peak/midpoint.

    d. Close the scan.

8. Repeat the **Z** and Ω alignment procedures until you are satisfied with the alignment.

9. On the top toolbar, select "**User Settings**", then "**Sample Offsets**" in the drop-down.

    a. Set the Current Position for both **2θ** and Ω to be zero.

    b. Click "**OK**".

Your sample should now be properly aligned.

4. Taking data

1. Start by ensuring the system settings are appropriate for taking data.

    a. On the "**Incident Beam Optics**" tab, set the Beam attenuator usage to "**At preset intensity**".

    b. Insert the desired **divergence slit** into the incident beam optics; e.g. 1/4°, 1/2°, etc.

    c. If you don't need the best possible resolution, it is recommended to remove the **0.18° parallel plate collimator** slit to improve signal intensity.

2. Next, generate a new program that defines your **θ-2θ** scan.

    a. On the top toolbar, select "**File**", then select "**New Program**" in the drop-down.

    b. On the pop-up, select "**Absolute Scan**" in the Program type drop-down.

    c. Click "**OK**".

d. On the next window, select "**2Theta-Omega**" in the <u>Scan Axis</u> drop-down.

e. Enter your desired scan parameters; i.e. start/end angles, step size, time per step.

f. Close the window using the red **X** button in the upper right corner. The **Data Collector** software will then ask if you want to save the program you just created. Save it in your personal program folder.

3. Finally, run your saved program.

    a. On the top toolbar, select "**Measure**", then select "**Program**" in the drop-down.

        i. Click the "**Browse**" button.

        ii. In the file explorer, select the file containing your previously saved program.

    b. On the next window, click the button with an icon that resembles a file folder.

        i. In the file explorer, select the location you want to save the finished data file.

        ii. Type in a filename, then click "**OK**".

    c. Type in any additional metadata you want saved to the file; i.e. ID, name, etc.

    d. Click "**OK**".

Your scan should now be running! Wait for it to finish.

5. Shutting down the system

1. Return the system to its standby settings.

    a. On the "**Incident Beam Optics**" tab, set the <u>Beam attenuator</u> to "**Do not switch**" and select the "**Activated**" checkbox.

    b. On the "**Instrument Settings**" tab, set the <u>Generator</u> to **40 kV / 20 mA**.

2. Remove your previously set offsets.

    a. On the top toolbar, select "**User Settings**", then "**Sample Offsets**" in the drop-down.

        i. Click "**Clear All Offsets**" and then click "**OK**".

3. Return the sample stage back to its home position (all motors at zero).

4. On the top toolbar, select "**Instrument**", then select "**Disconnect**" in the drop-down.

5. You can now close the **Data Collector** software.