

Operating Instructions for the ARL Equinox 100 XRD

I. Start Up

1. Check that the emergency stop button is released.
2. Push the white button marked "Power ON" located on the front panel.
3. After powering the instrument ON, **the red light of the "Power OFF" button turns ON.**
4. After several tens of seconds, the fan will turn ON to cool the instrument and the system is ready for operation.

Note: The ARL EQUINOX X-Ray diffractometer needs to stabilize for about thirty minutes after starting the instrument. This time is necessary for the best performance of the instrument.

5. Check that the bubbler located next to the instrument on a universal stand has ~4ml of distilled water.
6. Turn the blue valve located on the back of the instrument (Figure 1) **VERY SLOWLY** to the Flow position (vertical). If not turn slowly the water in the bubbler will shoot out.
7. Check that gas flow is about 7-10 bubbles per minute. If the flow is either lower or higher, please contact Dr. Ponce.



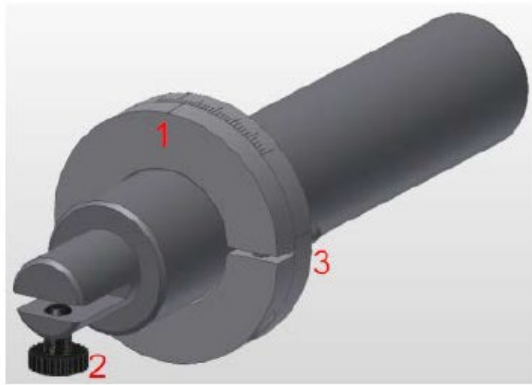
Figure 1. A portion of the back-right side of instrument (left image) showing the location of the gas flow valve (right image).

II. Software Controls

1. Log in the computer using your credentials.
2. Software Icons available:
 - **InelServers:** Establishes connection between the XRD and the PC.
 - **CpsClient:** Is used to turn on the detector
 - **Generator:** Controls the X-ray tube
 - **AcqCps:** Is the acquisition software
 - **Match!:** Is the data analysis software
3. Click on the **InelServer**, wait a moment, and then click on the **CpsClient** icon
 - Follow the prompts on the screen
 - Check that the gas pressure light is green (between 5.75 - 6.0). If the pressure is out of range please contact Dr. Ponce.
 - Click on High Voltage button to turn it on if not green. The final value should be around 9900V. The value should eventually stabilize.
 - You can close this window now
4. Click on the **Generator** icon
 - The initial values should be 1KV and 0mA
 - Click on the Xray button it should go from dark red to bright red when on and the X-ray sign in front of the instrument will lit up
 - Click on the value field, a dialog box will pop up and enter 20kV and 0.3mA
 - Wait for 2 minutes
 - Enter 40kV and 0.9mA
 - You can close this window now

III. Sample Holders

The EQUINOX 100 has a basic non-spinning sample holder suited for powder measurement in reflection and transmission modes (Figure 2). This sample holder is necessary for adjustment and alignment and can be used for measurement as well.



1 - Indicator of the incidence angle Ω

2 - Sample clamp

3 - Positioning pin

Figure 2. Non-spinning sample Holder

The non-spinning sample holder is designed to be easily used. The sample holder is equipped with a positioning pin to avoid any position mistake, an incidence angle indicator to read the incidence angle and a sample clamp (Figure 3) to maintain the powder sample mount.

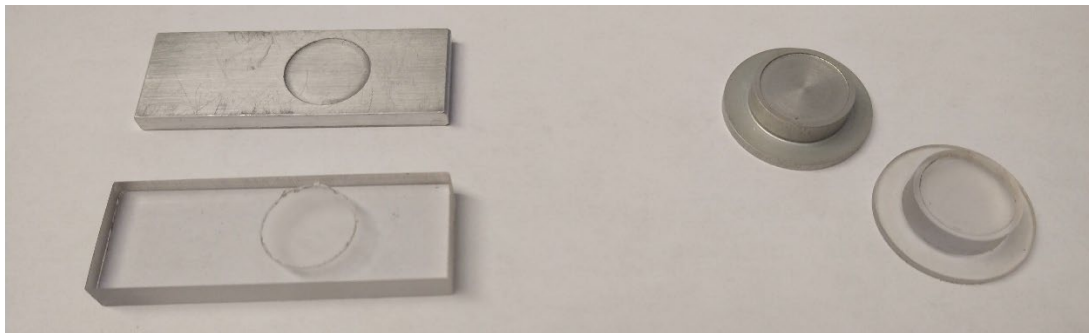
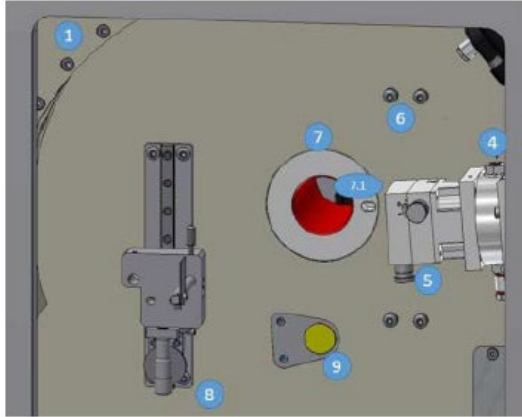


Figure 3. Sample claps for the non-spinning (left) and spinning (right) sample holder.



Insert the sample holder in the sample holder mounting hole of the instrument (7), do not use any unnecessary force. The sample holder and the mounting hole (\varnothing 30 mm) are designed to avoid any mechanical clearance resulting in irreproducible results. The pin of the sample holder has to be inserted in the reference hole (7.1).

The incidence angle indicator consists of two rings. One ring is fixed while the second one is free. This allows the sample holder to rotate in order to change the incidence angle. This second ring can be tightened to avoid changes of the incidence angle. The way of reading the incidence angle indicator is described in Figure 4. The longest line represents the incidence angle equal to 0° . Each short line represents 2° of incidence angle. The medium long lines represent 10° of incidence angle. The indicator then serves to read the total incidence angle.

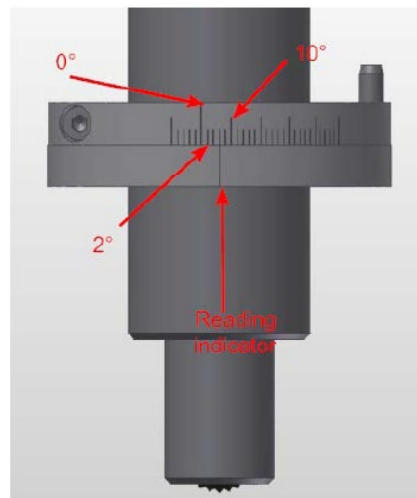


Figure 4. Incidence angle indicator. In this configuration, the incidence angle is equal to 6°

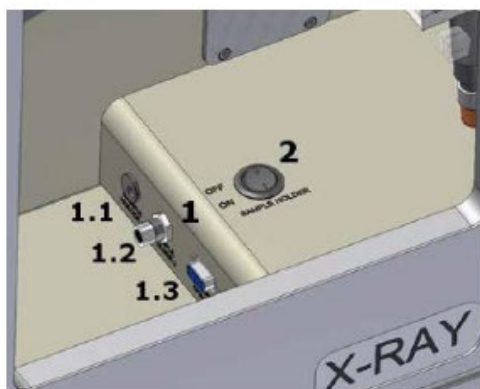
In addition to the non-spinning sample holder, the EQUINOX 100 has a spinning sample holder (Figure 5). The spinning sample holder is also designed to be easily used. The sample holder is equipped with a positioning pin to avoid any position mistake, an incidence angle indicator to read the incidence angle, a spring-loaded sample clamp to maintain the powder sample mount (Figure 3), and a cord/plug to power the spinner motor.



Figure 5. Spinning sample holder

The insertion of the spinning sample holder in the sample holder mounting hole of the instrument and the adjustment of the incident angle indicator are the same as for the non-spinning sample holder.

The power supply plug for the spinning sample holder is located below the sample area.



1. Set of plugs to connect the sample holder.

1.1 A jack connector for the 12V power supply for spinning the sample holder.

2 The switch to turn ON the sample holder power supply.

Figure 6. Spinning sample holder power supply and On/Off button.

The spinning sample holder uses a round sample mount (Figure 3). To load the sample mount onto the spinning sample holder, follow these steps:

- Turn on the sample holder power supply so the opening is pointing to the left.
- Rest the edge of the sample mount and at the same time push down and forward until the sample mount is all the way in.
- **Please practice several times with an empty sample mount first so you won't drop your sample in the instrument.**

IMPORTANT: Both sample holders need to be aligned to have an incident angle of 6 to 7 degrees.

IV. Sample Preparation Procedure (for spinning and static sample holders)



In order to prepare your sample, you will need a powder mount, a spatula and a glass slide (as used for microscopes). If the powder to be measured is too coarse, it's necessary; to grind it a bit using a small mortar.



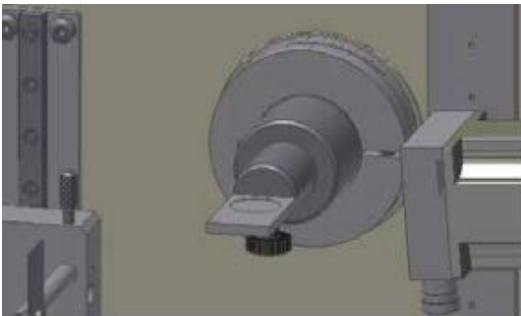
Fill the cavity with the powder.



Use a glass slide to make the surface flat and homogeneous.



The surface of the powder must not exceed the level of the surface of the powder mount, in order to avoid line displacement error due to height error.



The powder mount is clamped in the sample holder by tightening lightly the screw of the non-spinning sample holder.

V. Data Acquisition and analysis

1. Click on **AcqCps** icon

- **IMPORTANT:** On the top-left corner of the screen the generator status is shown. Check that the KV and mA values of 40 and 0.9 respectively are shown and that they do not change while using the instrument. If they change go to generator and reenter them. If this failed restart the instrument.
- Set Preset Time to the time you want to collect data for in seconds, it will turn from red to black by pressing enter.
- The above parameter is the only one needed to be altered when running a sample, the rest are fixed and related to the type of detector the instrument has.
- With sample in the instrument press the Start/Stop button
- If you think you need to collect data for a longer time just enter a new preset time and run it again. The data acquisition will be summed to the previous one.

Example: You ran it for 30s and you want to run it for 30s more. Then you will enter 60s for the second run. The instrument knows you already ran it for 30s, so it will just run it for 30s to make a 60s total run.

- More details about using the AcqCps acquisition software can be found in Appendix A.

2. Saving your data

- Once you have completed your acquisition, save your data by clicking on the DAT icon at the bottom of the screen. If you want to export your data, there are several options at the bottom of the screen, but it is recommended that you export it as an ASCII file since it is recognized by most graphing/spreadsheet software. To do this just click on the TXT icon.
- **IMPORTANT:** Before running your next sample click on the CLEAR button.

3. Data Analysis

- To perform your data analysis, you will need to open the MATCH! Software from the Desktop and open/import your *.dat file.
- More information on how to use the software can be found in the MATCH! Manual provided and in the videos bookmarked in the Chrome browser.

Phase Identification Procedure:

- On the option menu under File, click on Open and select your *.dat file
- You may want to cut/select the 2Theta range to perform phase identification. Place the cursor over the left-Y-axis until it turns into an arrow/scissors icon, click and drag until 10-20 degrees. Now repeat the previous procedure on the right-Y-axis and cut to about 110 degrees.
- On the Option Menu under Pattern, click on Peak Searching
- If a peak is missing press Ctrl+Right Click

- Under Pattern select Profile Fit and select Fit FWHM, then Fit Intensity, then Fit 2theta, and then Profile Fit. You can also just press F8, F7, F6, and F5 from the main screen.
- On the top-right panel of the screen under the composition tab, click on Toggle to unselect all elements (they will turn all red)
- Click on Optional (yellow) under “Element selection by mouse”
- Click on the elements that you know or suspect are present in your sample (they will turn yellow)
- Click on the Run Phase Identification icon (yellow diamond w/a green dot) on the top of the screen.
- The search results will be displayed on the bottom-left panel, listing from the top the best matching results (Large FoM value). The ICDD Cards only contain peak values up to 100 degrees.
- You can use the up/down arrows to scroll through the pattern match list.
- Double click on what you think is the best matching result and it will display its ICDD Card information on the bottom-right panel.
- To see the ICDD Card information click on the Data Sheet tab, or Peak List tab to see how well the peak match.

VI. Shut Down

When the X-Ray diffractometer is not used for more than an hour, we advise to run the generator at a power saving mode, 20 kV, 0.2 mA.

When the instrument is not used for more than one day, it can be shut down completely.

1. Reduce the generator power to 0 kV and 0 mA.
2. After the generator has reached 0 kV and 0 mA, quit all the software and terminate the communication between the instrument and the computer.
3. Push the red button labeled "POWER OFF" located on the front panel. After you pushed it, this red button will turn OFF and the white button "POWER ON" will illuminate.
4. Turn the blue valve located on the back of the instrument (Figure 1) to the OFF position (horizontal).
5. Log off from the computer

VII. Emergency Stop

In case of any problem (electrical, radiation or mechanical) there is an emergency stop button located on the front panel of the instrument. Pushing the emergency button immediately removes the power to the instrument, including the X-Ray generator. Be aware, that pushing the emergency stop button will result in communication loss between the control computer and the instrument.

CAUTION! The emergency stop should not be used for routine shut down of the generator since repeated rapid power shutdowns may dramatically reduce the lifetime of your X-Ray tube and damage the X-Ray generator.

Appendix A

(caution some features may not apply to our current version)

Basics of running an acquisition

We assume now that your X-Ray generator and detector are switched on and that your sample was carefully prepared in a sample mount and set in your spinning sample holder. If you have a 4 circle goniometer with a Phi rotation equipped with a stepper motor, please refer to section Moving on page 28.

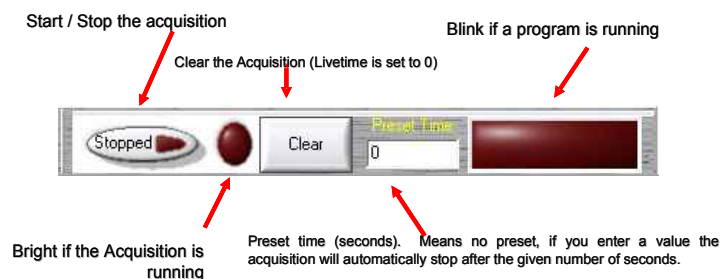


Figure 17 : Acquisition tools toolbar

Since the previous acquisition data are stored in the memory buffer, your acquisition screen is maybe not blank. Thus, before starting an acquisition you have to clear the memory by pressing the *Clear* button (see Figure 17). You'll see that your livetime is also automatically set to zero. Note also, that you may clear a running acquisition without stopping it; it will start again from the beginning.

Open your shutter, and start the acquisition by pressing the start/stop button . To stop the acquisition press again on this same button.

At this stage we did not use the preset time functionality. If the preset time value is zero, this means that this is not in use, thus the acquisition will go on, until the acquisition is stopped manually (or by a running batch experiment). Now, if you want that the acquisition is automatically stopped after a given acquisition time, you may change the preset time value.

Stop the current acquisition, to do a 20 minutes acquisition of our sample, enter 1200 in the preset time edit box (**preset time is in seconds**) and press enter to validate. Then start again the acquisition, it will be stopped automatically after 20 minutes.

At the end of the acquisition your pattern should look more or less like the one shown on Figure 19.

During the acquisition you may use the display tools to test the graphical features of AcqCPS.

Scale tools

To zoom in simply move your mouse from the top left region you would like to enlarge down to the bottom right while pressing the left mouse button. To zoom out, double click on the data display, or while pressing the left mouse button go from bottom right to left. To pan right click while moving the mouse.

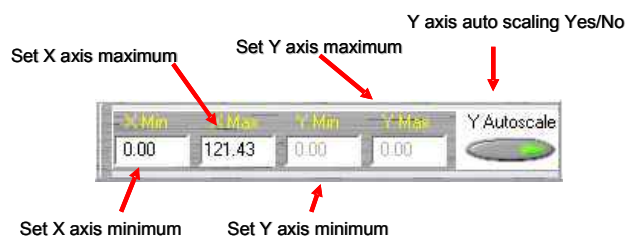


Figure 18 : Scale toolbar

You may also define a fixed display region manually by entering minimum and maximum values for the X and Y axes (see Figure 18). Note that to have access to Y axis limits, the Y autoscale feature as to be switched off.

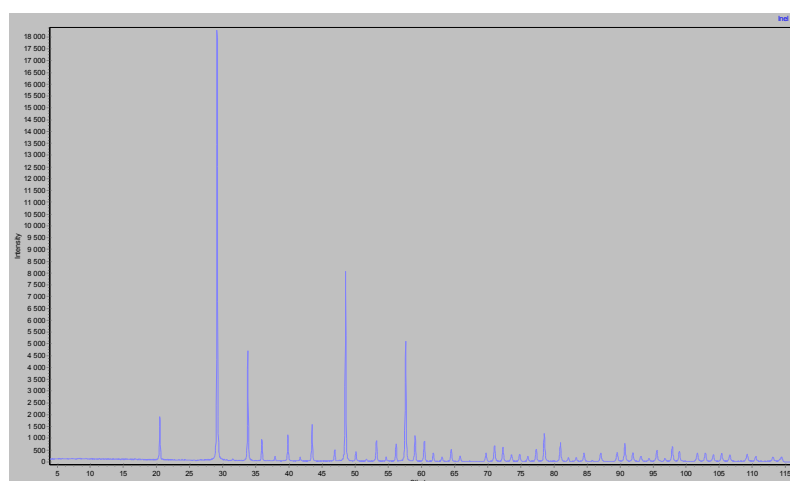


Figure 19 : Yttrium oxide pattern obtained in 20 minutes using a copper tube, Germanium flat monochromator and a CPS 120 detector

Superimposing patterns

With AcqCPS you have the possibility to overlay previously acquired patterns to the current acquisition.

You may overlay DAT files (classical Inel data files). If you want to overlay data contained in an FDT file, you have to save the scans you want to overlay as DAT files using IMAD.

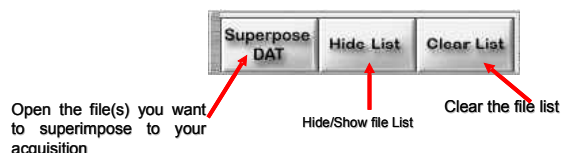


Figure 20 : "Superposition" toolbar

Click on the *Superpose DAT* button and select as many DAT files as you want. As an example you may overlay the data from the "Yttrium oxide" file contained in the example folder of your Symphonix CD.

A list will appear that shows the current data set in memory that can be displayed (see Figure 21). You can move this list by dropping its edge with the mouse. The overlaid data use the right axis as Y axis and top axis as X axis to arrange for differences in acquisition time.

To remove temporarily a diagram, unmark the checkbox near the data filename. To change the colour, double click on the colour mark between the checkbox and the name.

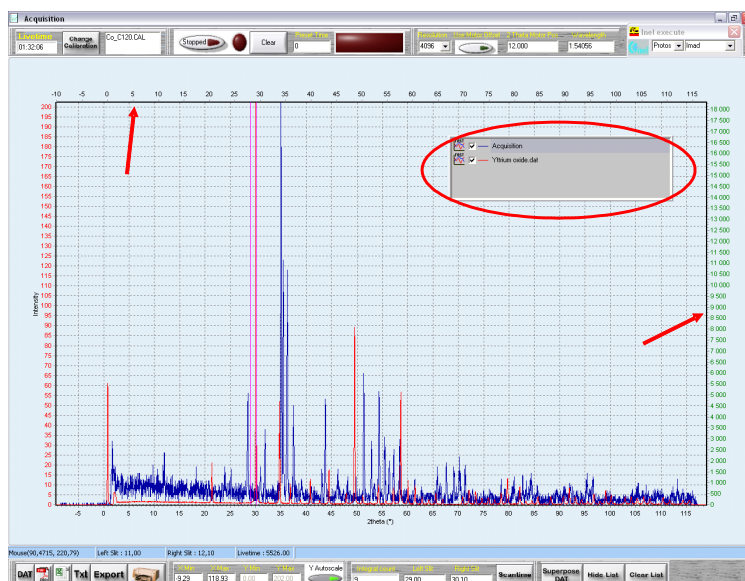


Figure 21 : data overlay example

To add additional data to the list use again the *Superpose DAT* button.

To hide the list, click on the *Hide List* button, to recover the list box, click on *Show list*.

To clear the list, simply click on *Clear List*.

Display tools

Additional tools are available that enables, beside others, to have access to 3D display functionalities . To enable the 3D display, click on the 3D display On/off button (see Figure 22).

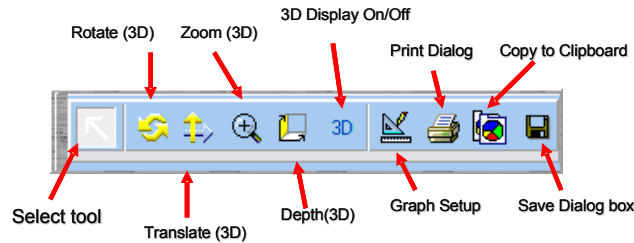


Figure 22 : display toolbar

To rotate, zoom in, zoom out, pan a 3D display you have to select the appropriate tool on the display toolbar.

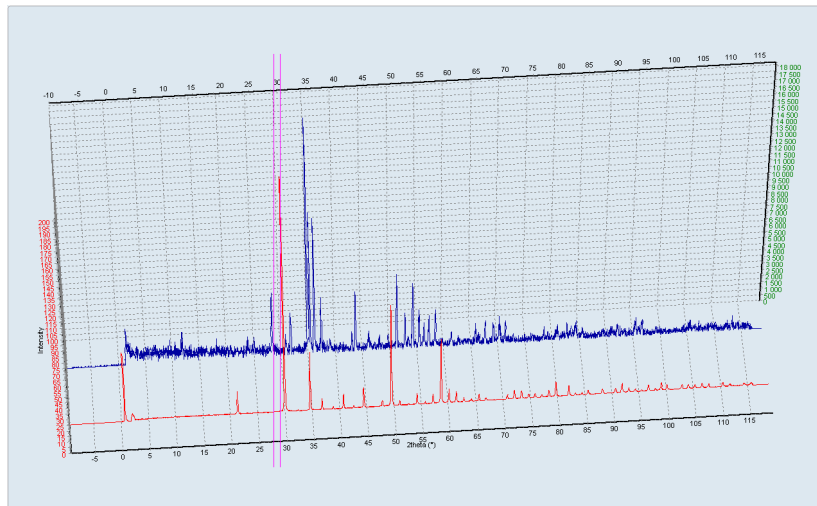


Figure 23 : 3D display example

Saving / Exporting data

In AcqCPS there are several possibilities to save your data, depending on what you plan to do with it. Most of these are accessible by using the Save / print toolbar (see Figure 24).

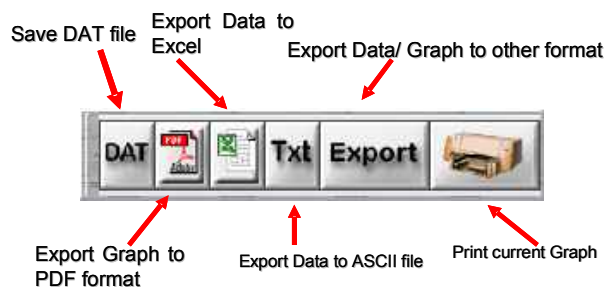


Figure 24 : Save / Print toolbar

To save your data to a DAT file click on *DAT* button. DAT files are standard Inel data files; they contain a lot of information, your diagram of course, recorded in channels but also the wavelength, detector calibration information, date, acquisition, motion position, information about your sample.... **In AcqCPS, this is the only file type that permits crystallographic investigations; you should always record your data in a DAT file.** The other available formats (ASCII txt, PDF, Excel) are there to help you to import your data in standard data analysis software or plotter (Excel, Origin, sigma plot...), they do not contain information about the XRD system used that are required for typical studies, like search/match, crystallite size determination, data corrections etc.

More, the step size of a CPS detector is not constant over the whole detector, we say that the detector as not a constant step size. This makes the PSD somehow unique compared to more classical scanning diffractometer with point detectors. Third party XRD data analysis software usually accepts only constant step data as ASCII files. If you save you data as Txt file in AcqCPS they won't be linearized (conversion to constant step data), and third party software will not be able to handle them because of that. You have the possibility to get constant step ASCII files from DAT files in IMAD, please refer to the section Import/Export Data on page 41.

With the same above mentioned restrictions (used only to use "data plotter" software), by clicking on export, you may export your data, as pictures or native data, to additional formats like bmp, or XML.

You can save a data file even before the end of an acquisition : in the DAT file, the acquisition time, date, and all other data contained in this file are the ones contained in shared memory when the file is written to the disk.

To Print your results, simply click on the printer icon. A dialog box will appear where you may select your printer, adjust your margins and more, like most other windows based software (see Figure 25).

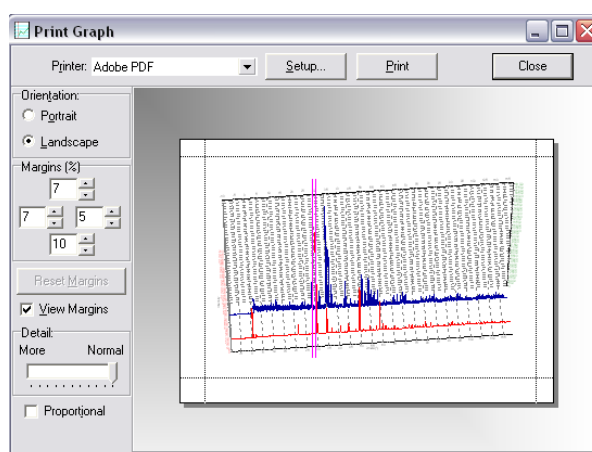


Figure 25 : Printing dialog box

Slit Definition

There are 2 purple lines on main display : these are the numerical slits. They are used to get the integral count from a given angular range, like we would do by closing/opening received slits in front of a point detector. In other words, it acts as a rate meter that counts only the photons between the 2 slits.

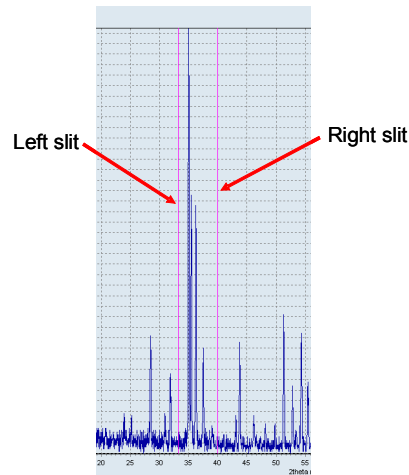


Figure 26 : both slits

You can move the slits with the mouse : to select a slit, put your mouse over it, you'll see that the mouse icon changes, and move the slit by keeping the left mouse button pressed. The integral count between the slits and both slits position is indicated on the slits tool bar (see Figure 27).

Figure 27 : Slits toolbar