

Powder X-Ray Diffraction (PXRD/XRPD)

Rigaku MiniFlex600,



NOTE: This machine produces ionizing radiation. Please take note of and read the local safety rules displayed and ensure you are supervised when using the equipment.

SAMPLE PREPARATION

The sample should be a fine powder with no large crystallites present. Large crystals should be ground using a pestle and mortar to give even sized particles in a powder form. If wet or in solvent, dry the sample as much as possible before grinding.

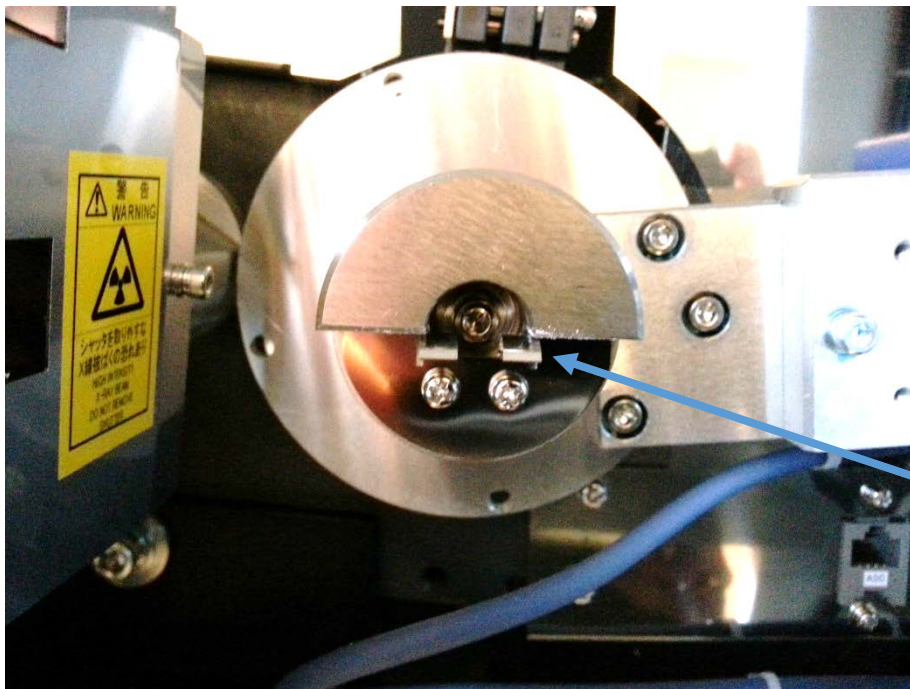
There are several options for preparing a sample, glass sample holders or an aluminium holder. The glass sample holders are recommended - as with all glass items they will break so take care when using them. There is a frosted rectangle covering approximately one third to one half of the glass plate which is indented on one side to give a small well for the sample to sit in (fawn coloured area as seen below).



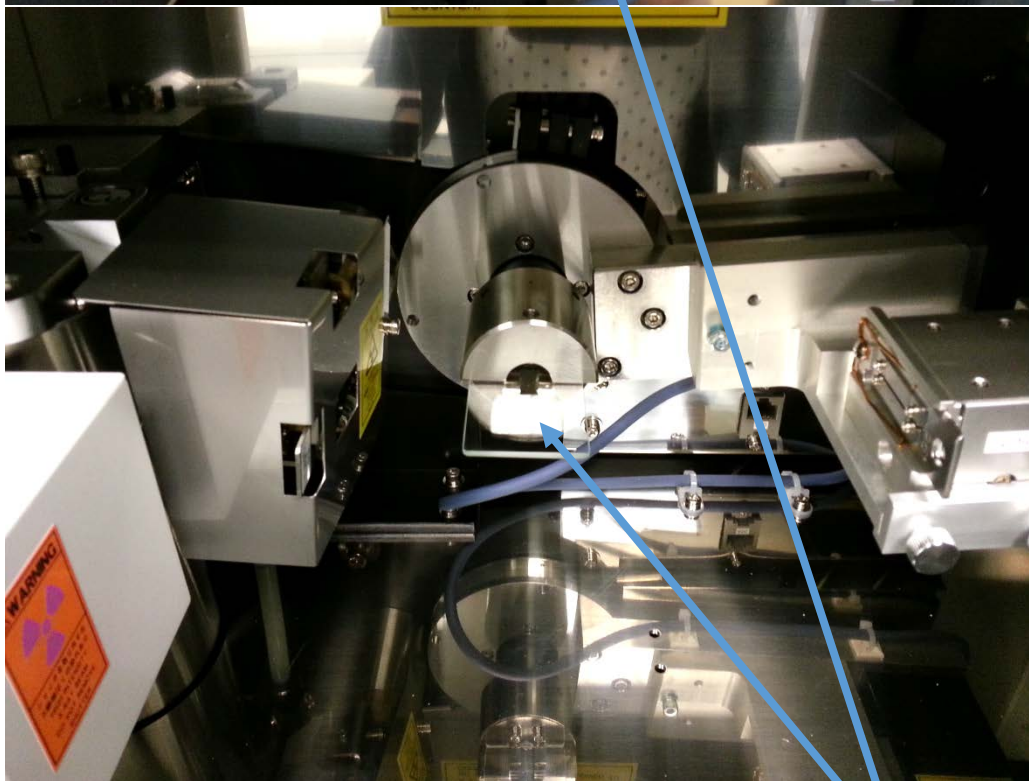
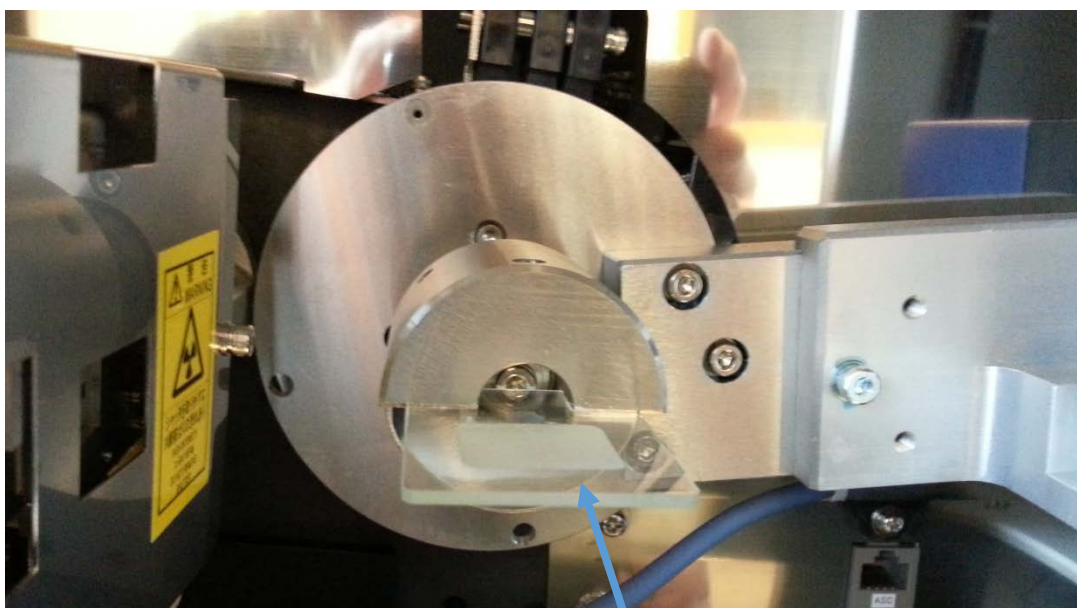
The sample should be placed in the well ensuring the correct side of the holder is used (one side is completely smooth, this is NOT the side you want). Carefully spread and flatten the powder using the edge of a glass slide to give an even covering. Use the face of the slide to gently press the sample making it level with the top plane of the glass and suitably compressed (no loose powder).



Open the door to the diffractometer and slide the sample into the space between the clips then close the door.



Sample is placed here, between clips and semi-circle seen



Sample holder in position

Before collecting data ensure the chiller unit (on the floor in front of the bench and computer) is switched on (you will hear it if it is) and turned off when the machine is not in use.



Door lock button. This will flash yellow and the machine will beep when the diffractometer is on and the door is not locked.

Operate – will be green/yellow when diffractometer is on and working. Red if there is a problem.

HV Enable – key must be in place

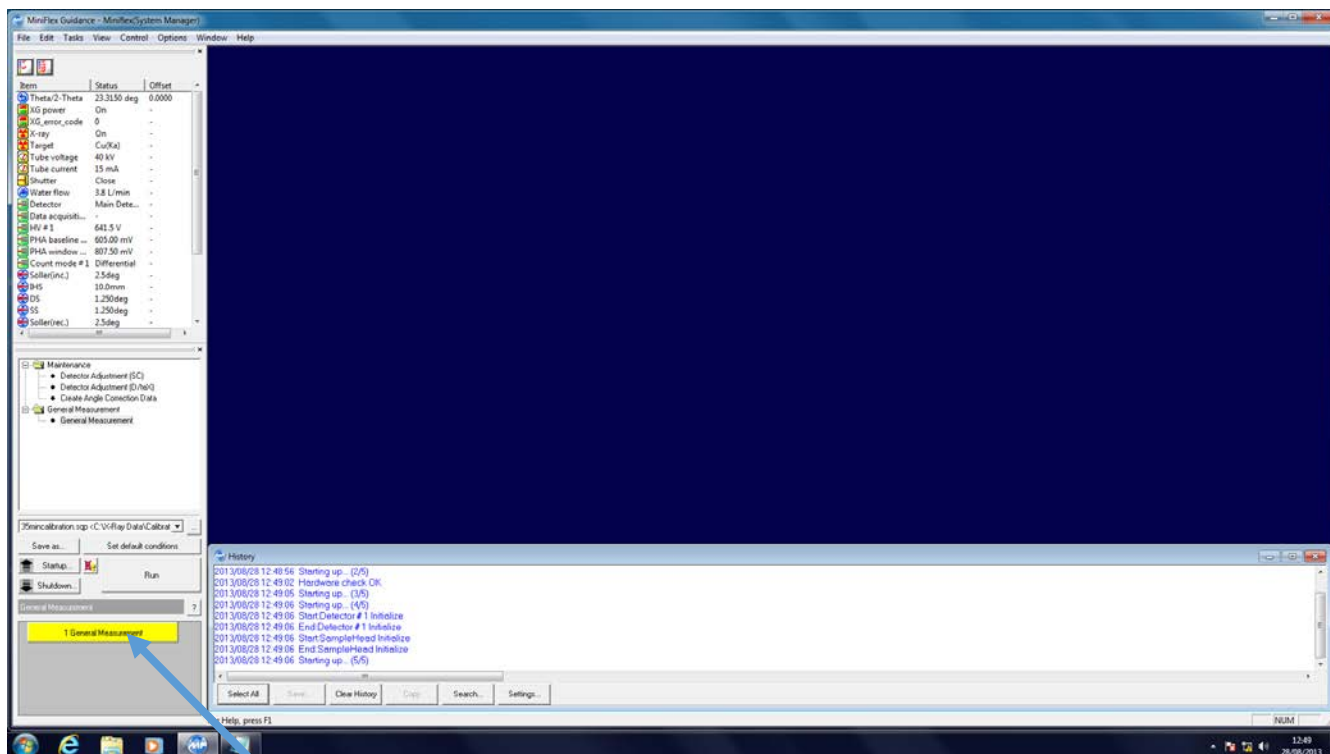
Power off

Power on

COLLECTING A POWDER PATTERN

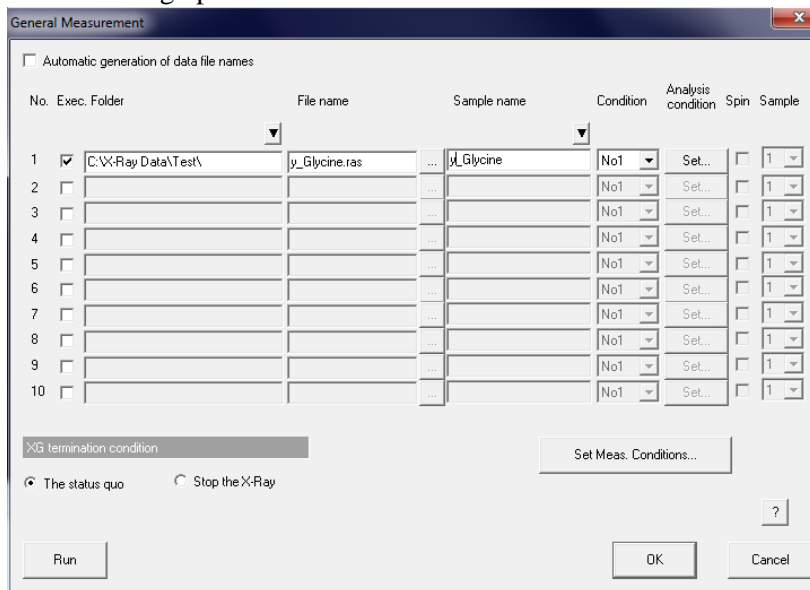
To run a sample:

- Turn the diffractometer on (if not already) using the green button and the chiller (below) using the black switch. The 'operate' light should light up and the orange light up on top of the diffractometer will be lit
- Lock the door (to stop the beeping) once the sample is in place
- On the computer open up MiniFlex Guidance (shortcut on the desktop), ensuring the diffractometer is on (so that the two can connect)
- Login with name 'MiniFlex', no password
- To collect a pattern click the yellow 'General Measurement' button at the bottom of the left hand menu



General measurement button

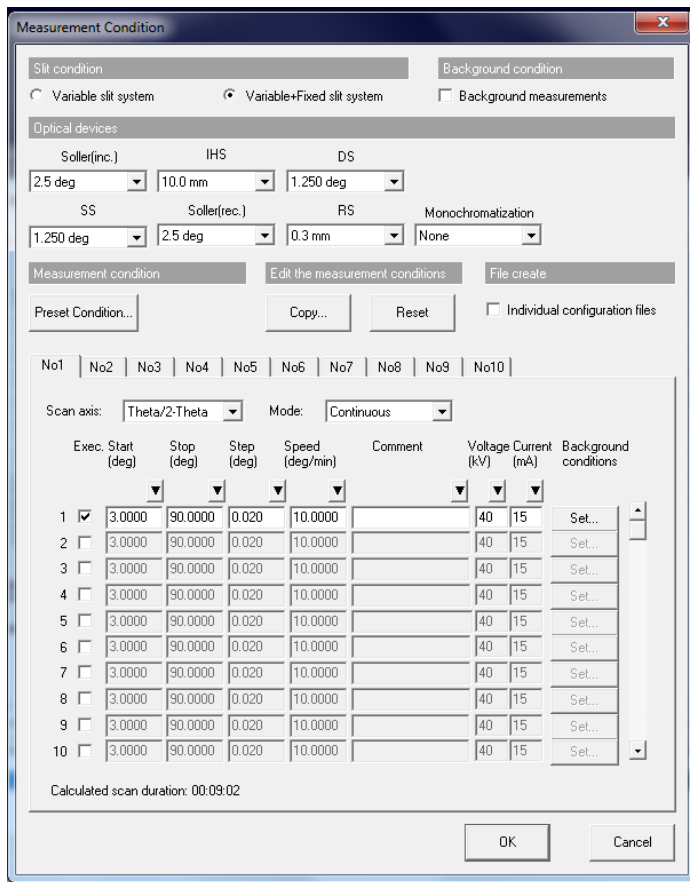
This will bring up a new window.



Use the ... button next to file name to browse the folder and set the file name. Save files in the Crystallography Advanced Practical folder (C:/X Ray Data/Crystallography Advanced Practical), file name following the structure:

INITIALS_SAMPLENAME_DATE_EXP

eg LM_yGlycine_1Oct13_2-50. You will also need to enter the sample name (same as file name). Select No1 Under condition and then use 'Set Meas. Conditions' to check the profile of the data collection.



Ensure the profile in row 1 is as follows and the Exec. box is checked for row 1:

Start (deg): 3.0000

Stop (deg): 90.0000

Step (deg): 0.020

Speed (deg/min): 10.0000

Voltage (kV): 40

Current (mA): 15

Then click OK.

When set up, click 'Run' to start the data collection experiment.

If a message box appears saying

'Install optical devices as follows: Soller(inc): 2.5deg |HS:10.0mm DS: 1.25deg

SS: 1.25deg Soller(rec): 2.5deg RS: 0.3mm

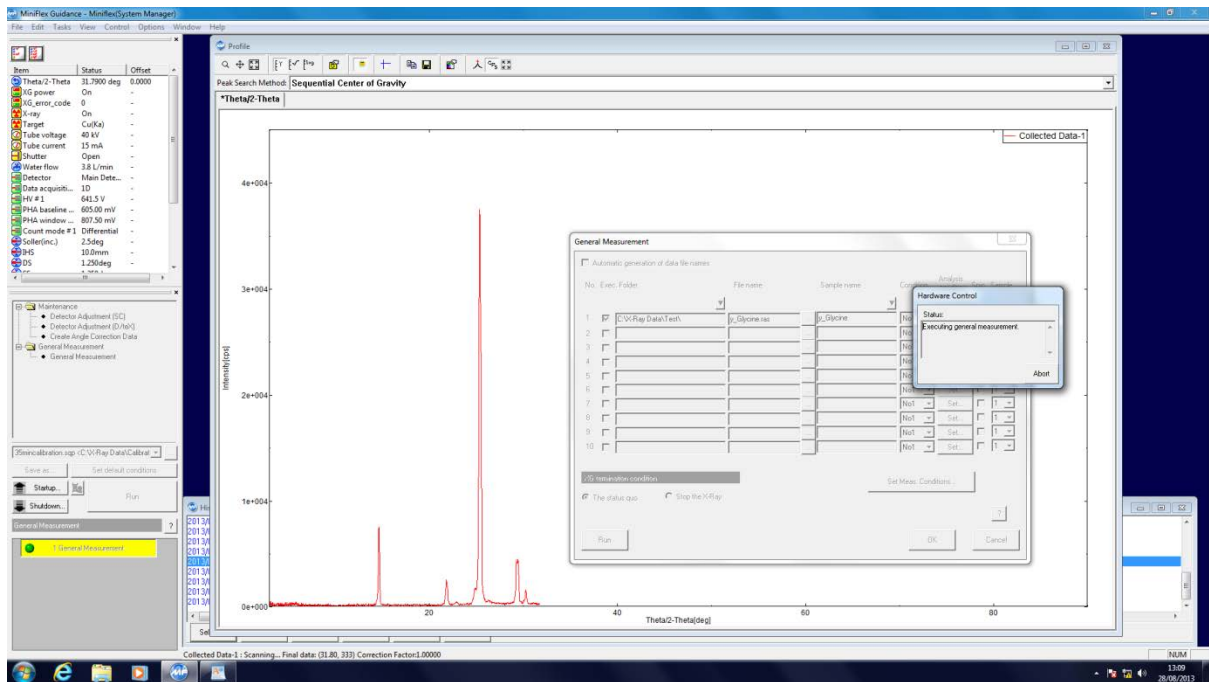
Filter: none

Monochromator: none'

Select OK and the experiment will start.

If error messages appear there could be a number of causes – most likely is the chiller is not on (flicking the on/off switch on the box will resolve this).

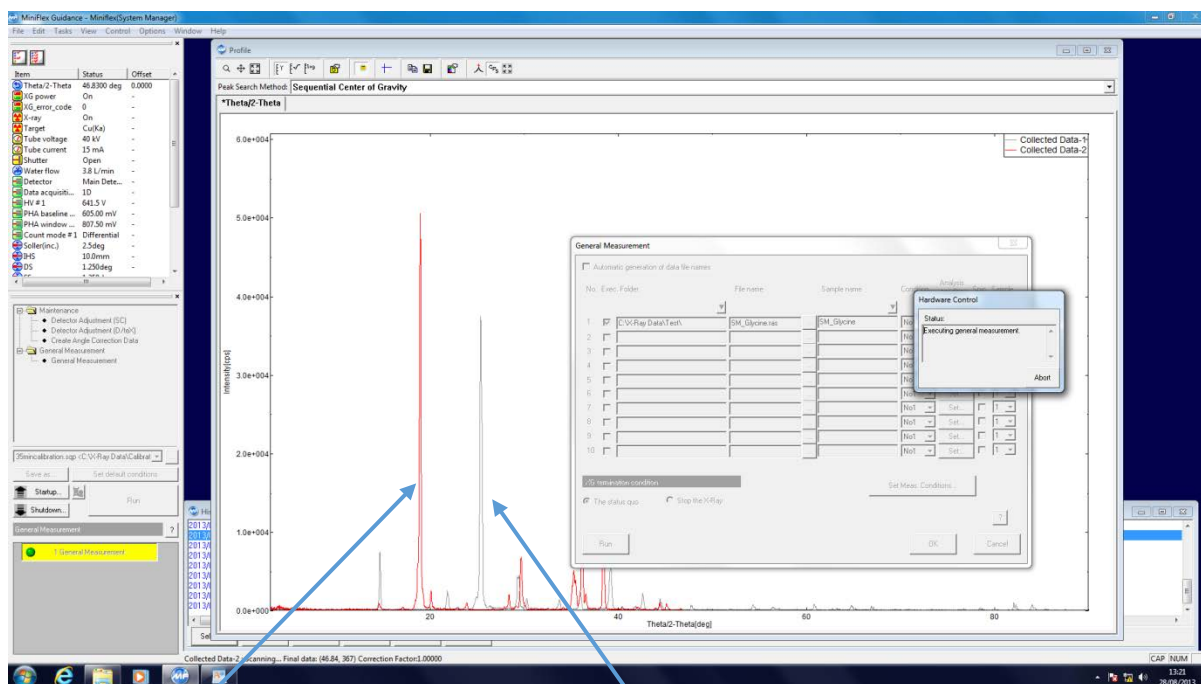
When the experiment starts a dialog box saying 'executing general measurement' will appear, the pattern will start to be generated and will be visible behind the dialog boxes in real time as it is collected.



When the data collection is finished the General Measurement window will no longer be greyed out and it will be possible to run another sample. The MiniFlex generates three files (.asc, .ras and .raw) however it is also possible to export the pattern to alternative formats when still displayed on the screen. To do this click File → Export and this will give a section of possible formats. X-Y (.txt) format will enable you to open the file in excel.

If you wish to run a second sample, before doing so fill in/change the details in the General Measurement window as appropriate (changing the file and sample names). Unlock the door and replace the sample.

The profile window displaying the pattern can be closed after the measurement is complete, or alternatively it can be left open. In the latter case, the next pattern collected will be displayed along with the original giving an overlay of the two (which can be good for sample comparison if required).



New pattern (red)

First pattern collected (grey)

When you have finished, ensure the sample holder(s) are fully cleaned, removing all traces of your sample from the holder and area surrounding the MiniFlex600. Please leave the area and equipment as you found them, ready for the next person/group to use. If no-one else is waiting to use, turn off the diffractometer (white button) and the chiller unit.

VIEWING AND ANALYSING XRPDS

The software will save three files for each pattern; a .raw file, a .ras file and an .ASC file.

To open and view these there are various programs available:

- 1) Crystaldiffract (opens the .ras file) available on all university computers (Your School Software → Chemistry → CrystalMaker → CrystalDiffract)
- 2) JPOWDER, a free download available from <http://www.jpowder.org/> (opens the .ras file and also .xye files)
- 3) Excel (if you have exported the file as an XY file)

You will collect three powder patterns, one for each polymorph and one of the starting material. You should look at each pattern separately and also overlay appropriate patterns to allow comparisons and differences to be noted.

Your observations as you are viewing and analysing the patterns should be noted, detailing the pattern you are looking at and whether you are looking at more than one or making comparisons to another. Include an overall summary of the powder patterns and what they tell you about the individual polymorphs and starting material.