SCXRD: Running a Crystal

XtaLAB mini

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.

You should use the microscope to inspect your crystal samples and select an appropriate crystal to run (see choosing a crystal) and ensure you do this before grinding up your sample for XRPD.

The XtaLAB mini controls (similar to MiniFlex controls)

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

Inside the cabinet

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 on

Power off
Axes of rotation

Monochromator (graphite)
Collimator
Video camera
Crystal goes here (goniometer head)
Cryostream
Fixed detector
RUNNING A SAMPLE

Ensure the all peripheral equipment is turned on

1. Turn on dry air (behind)
2. Turn on pump (behind)
3. Turn on power (at front)
4. Open valve fully

The cooling water also needs to be turned on (button on front).

All of these should be on before the XtaLAB mini is turned on (green button on front). This will cause the door lock button to flash and intermittent beeping to be heard. Press the door lock button to stop this.

Cooling Stream
To run samples below room temperature the desktop cooler is used to create a cold stream which passes over the crystal and cools it. Ensure all the above has been turned on for at least 20 minutes before starting cooling.
To start the cooling, open up the Cryopad DTC from the desktop

This will open up the following window
If the cooler unit and pumps etc have not been turned on there will be a problem connecting to the Desktop cooler and the left hand side will have a red icon with ‘Not Connected’ instead of the green tick and ‘Connected’. If there is a problem, shut the window down and check the desktop cooler is on appropriately before restarting the Cryopad software.

To set the temperature enter the value (K) in the ‘set temp’ box (default 170K and this is the lower limit of possible temperatures). When ready, click on ‘start’ and the cooling will start. It will take some time to cool down to 170K and the ‘Gas Temp’ value will show the cooling progress and current temperature.

The taskbar icon will also be generated once the cryopad software has been started.

To run a crystal:
Open Crystal Clear Auto 2.0 software from the desktop and login
Username: student, no password.
You may need to open a new sample or project (new sample if you already have a project, ie this is not the first crystal you have run, or a new project if you have never run a crystal on this machine before). File → New Sample or New Project.

The open dialog box (Open Sample) will require you to choose your project and sample name. Tick the ‘new project’ box) and type the project name, then do the same for the sample. Note samples (eg crystals) must have different names but there can be multiple samples in one project.

Project name: AdvPrac_INITIALS eg AdvPrac_SCLM
Sample names (must be unique): YEAR_INITIALS_SAMPLE, eg 2013_SCLM_001

Then click OK and the instrument will initialise and you will see the Goniometer values changing.
When fully initialised, the values should read $\omega$ -70.0, $\chi$ 54.0 and $\phi$ 0.0 and the flow bar on the left hand side will progress to mount crystal, with several dialog boxes opening (second screen shot)

**NOTE**, for all dialog boxes which open during the following process, NEVER press save. Always ‘run’ or ‘close’ each window.

You will need to mount the crystal onto the goniometer at this point. Press the unlock door button, and wait for a pop up to appear on the computer screen (this will only appear if the software is open, if it is not the door will unlock at this point). Press ok when ready to open the door and the beeping will change from continuous to intermittent and you will hear the door unlock. The door can now be opened.
The inside of the diffractometer cabinet is shown earlier detailing the different components. Be
careful not to accidentally move any components from their set positions when working. The
goniometer will be in the mount position as shown below, enabling access to it. If you already have
your crystal mounted on a goniometer head, place this onto the mount and tighten the screw
(removing the head if the previous one is still in place). Alternatively you will need to remove the
head, mount your crystal on this and replace the head with the crystal on.

Once the crystal is affixed it will need to be centred to ensure that in any position (change of angle)
the centre of the crystal remains in the same place. To do this, look at the video image feeding to the
computer. You will see cross hairs (axes) on the display and an image of the crystal. You need to
move the crystal so that it is placed in the centre of the cross hairs when rotated on its axes (red cross
below).

To adjust the position of the crystal, you will first need to unlock the phi axis. Use the tool to unlock
as shown below (one small turn is sufficient).
You should get your crystal located as shown below, and when rotating the phi axis it should remain centred on the cross hairs. To do this, rotate the phi axis in 90° increments and adjust. The height may also need altering, to do this use the grub screw then rotate the knurled collar to bring the crystal ‘in and out’ of the view you have. Once centred, lock the phi axis using the lock screw at the bottom and close and lock the door of the diffractometer. There is now no further need to open the door until you need to change the crystal for a different one.

Using the video dialog box, you can switch between views inside the cabinet using the video tab, select capture device and then there are two options. One shows the crystal position and the other gives an image of the inside for monitoring the experiment if needed.
In the video view, under the view tab the ruler can be selected and the crystal dimensions can be measured by dragging the ends of the double headed arrow to the edges of the crystal. To rotate the crystal to a different view (to get the third dimension) use the Instrument menu at the top of the screen and select ‘Manual Instrument Control’. You will need to rotate in the phi (ϕ) axis by 90°. This should rotate the crystal and allow the measurement of the other dimension of your crystal.

In the ‘Setup and Mount Crystal’ window (already open), you will need to enter the dimensions of your crystal, the appearance of the crystal (from drop down menus) and the molecular formula (using capital letters).
When filled in as appropriate click ‘Run’. The x-rays will then power up and the orange light on top of the XtaLAB mini will turn on.

The status in the instrument status display will change to ‘collecting’ and the shutter status will be seen to open and close.
Once the first image is taken, it will appear on the screen.

Good diffraction will give spots which cover over half to two thirds of the detector image originating from the top of the view shown. Select Resolution Arcs and in the pop up window check the box at the top next to ‘visible’. This will display red arcs which can show how good the diffraction is. If spots are seen out to the 3rd band the diffraction is reasonable and structure solution will be possible. If diffraction is seen out to the 4th band (or further) then the diffraction is really good. Anything which does not have spots extending into the third band, or has poor intensity, could prove problematic in solution.
The estimated time to finish will be shown in the bottom left, and the image number (of 12) displayed on the right in the grey panel. When all images have finished collecting the flow will follow through to predicting the spots. A pop up window will appear (Auto Cell Determination) showing the unit cell that has been found for the structure from the initial images. In this box the exposure time can be altered if needed. This should not need to be changed however increasing the exposure time can give greater intensity of diffraction if the initial images appeared with poor intensity. At this point you must also decide if this is the crystal you wish to proceed with (it is not a twin and of sufficient quality).

These two buttons allow you to switch between images collected to look at the diffraction spots. The blue spots show the spots which would be expected to be seen with the given unit cell. Browse through the images and see how well the diffraction spots collected and predicted match up.

Press collect and the data collection will start giving the pop up window shown below. This will show the time expected for the data collection and an estimated end time.
Click on the arrow under ‘Scale and absorption’ in the flow panel on the left to ‘block off’ the flow.

When the data collection is finished, click ‘integrate reflections’ and one run the flow bar will then progress onto ‘scale and absorption’, giving a final pop up window displaying graphs. The location chosen initially (for the project) will contain the output files for the data collection (shortcut to student data on the desktop).

**PROCESSING THE DATA FROM THE DIFFRACTOMETER**

Before you can solve the structure from the data files given by the XtaLAB mini you must process the .hkl file to give an .ins file. To do this, use Xplain (on the XtaLAB mini computer). Input the reflection file (the shelx.hkl file from the diffractometer) and the unit cell (the .cif file from the diffractometer). Step through the tasks on the left hand side ensuring the information given on the
right is as you would expect (given the unit cell you obtained from SCXRD and the knowledge from your literature searching). Generally, things in green are correct, or what the computer thinks should be the right options. You should look at the options given and think about the symmetry and cell parameters. In general you should choose the highest symmetry that is sensible. Follow through to the end and write both files giving them a new (but the same) name (to indicate these are post-processing files). Upload the newly written .ins and .hkl files to your LabTrove notebook for use later. You can also upload the crystalclear.cif if you wish as this contains information regarding the data collection.