

R-AXIS IV++ Hardware Documentation (User guide)

Rigaku RU-H3R Generator

Startup from cold

Air Conditioner.

Make sure that the air condition unit is on. If the unit is not powered up (no red light on the lower RHS of unit) it needs to be manually powered on by pressing the button on the lower RHS of unit. If necessary the temperature can be reset using the remote control unit (should be set to 18°C).

Chiller water.

1. Make sure that the white arrow on blue in-line water filter pack is pointing to the “filter” position, and the bypass red valve located in the pipe above the filter is “closed”.
2. Check that the flow meters behind the generator are registering flow. If not check to see if the red valves on the 3 inlet and outlet pipes are open. Fully open red outlet valves (located to the RHS of flow rate meters) before fully opening red inlet valves (located below flow rate meters). If there is still no flow, call the X-ray manager.
3. If the generator trips due to “CW” alarm, it is probable that the flow rate has dropped due to the filter getting clogged (check to see that the flow rate meters are registering some flow. Otherwise the problem is elsewhere, and you should get the X-ray manager). **To change the water filter**, open up red bypass valve above filter, making sure that the pressure on the inlet valve does not exceed 3.0 bar. Move white arrow to “off” position using the blue plastic spanner tool. Screw off the blue cylindrical filter holder. Make sure you have placed a bucket under the filter. Remove filter and clean holder. The filters used are wound polypropylene filters to remove 25µ particles. Soak the new filter in water for a few minutes. When screwing the holder back in place, try to keep the water level to the brim of the holder to minimise air introduced into the system. If pressure is above 1.8 bar, close down bypass valve to 1.8 bar. Move arrow to “on” position. Fully close bypass valve.

Generator power up

1. Turn on the isolator for the 3-phase power supply. This is situated behind the generator on the wall. The step-down transformer in the LH corner of the room should start to hum. If not, the switch on the main distribution board may have tripped off. This is in the locked junction box by the window in the R-axis IV++ enclosure. Don Akrigg and the building superintendent have the key (#16, yellow tag). Make sure the isolator is switched off, try switch back on the appropriate fuse and try turning on the isolator behind the generator again.
2. Turn on the main power switch at rear of the generator, near the floor, RHS. Then press the white power “on” button on the front of the generator below the red “emergency stop” button.
3. Check that five green LED's are lit. These are situated at the rear right hand side of the generator near the floor. These are marked
TEMP - goes out when water temperature to target outside specified limits
LW - goes out when there are water leaks in cooling system
HP - goes out when the water pressure at the target is greater than 3kg/cm²
TUBE FLOW - goes out when water flow to tube below 7L/min
TARGET FLOW - goes out when water flow to target below 14L/min.
If either of the flow lights is out, adjust inline valves before flow meters (see “in-line water filter” section above).
4. Exhaust is performed automatically by simply pressing SYSTEM START on the vacuum panel at the front of the generator. The system controller sequentially turns on the rotary pump (the RP LED on the vacuum panel at the front of the generator will come on), then about 20 seconds later turns on the

TMP (when the TMP is at full speed, the TMP LED on the vacuum panel at the front of the generator will illuminate). About five minutes later the ion gauge will come on (the IG LED on the vacuum panel will be illuminated) and digital readings (red) will appear (upper RHS, front of generator). When the vacuum gets below 10^{-4} Pa (approx. 150mV on the multimeter), the operated LED on the vacuum panel will come on.

5. When the multimeter reads approx. 100mV or less, the X-rays can be turned on. Check that the shutter open/close switch for the right hand port is set to external (EXT). Press the power ON switch on the X-ray panel on the front left of the generator. This supplies power to the X-ray generator controller, illuminating the power LED. The fine focus LED should also be on. Switches and LED's are inoperative for a period of five seconds after power on.
6. Press TARGET ON on the X-ray panel. This starts anode rotation. The READY LED on the X-ray panel and the green ready lamp on the post both come on, indicating that X-ray turn-on is achievable. Anode rotation should be clearly audible. The ion gauge reading will go up, so wait for it to come down again before proceeding further (reading below 100mV).
7. Before X-ray generation can proceed the R-AXIS controller must be on and power must be going to the radiation enclosure safety interlock mechanism. (See later). Press the X-ray ON switch on the X-ray panel. This starts X-ray generation by applying the minimum load (20kV, 10mA). The X-ray lamp will come on and the READY LED will go off. Similarly, on the post, the green ready lamp will go off and the red warning lamp will come on, indicating that X-rays are being generated.
8. Wait for the vacuum reading to drop below 100mV. The powering up of the generator can be controlled by the Xg program on the PC. Launch the Xg program by clicking on the icon (second from the bottom LHS above the CrystalClear icon). Pull down the task menu. If the filament has been replaced by the X-ray manager, it should be aged according to the "Age new filament" task. Select this task and the computer will automatically control the ageing (takes about 5 hours). If the filament does not need ageing select the task "Power up to 48kV 100mA" and the computer will automatically control the power up (takes about 3 hours).

Make sure that there is helium flowing into the Osmic Optics before opening the shutter (see Osmic Optics).

Shutdown

1. In the Xg program on the PC, pull down the task menu. Select "Power down X-rays" and the computer will automatically control the power down.
2. Turn off the X-rays by pressing X-ray off button on the X-ray panel. This will simultaneously turn off the GT and stop anode rotation. Continue with system exhaust for at least 20 minutes to let the target cool down.
3. Press SYSTEM STOP on the vacuum panel to stop system exhaust. The vacuum controller sequentially stops the TMP and then the rotary pump. Continue with cooling for at least 10 minutes to allow the TMP to cool.
4. Push the black power button on the front of the generator below the red emergency stop button. Switch off the main power switch at the rear of the generator, RHS near the floor.
5. Turn off the isolator for the X-ray generator 3-phase power supply.

Helium Supply

Helium floods the Osmic Optics to reduce adsorption of X-rays by air and to prevent oxidation of confocal surfaces. Cleaning of surfaces costs £1000's. **Do not therefore allow He cylinder to become empty.**

Changing Helium Cylinder

1. Turn off HE beam path flow control (HBPFC).
2. Close valve on He cylinder.
3. Replace cylinder with new one from stores (storeman has key). You will need to have filled out a stores order form.
4. Open valve on cylinder.
5. Adjust He regulators, if necessary, until they deliver flow rate (keep below 1.0 bar).
6. Set HBPF to slow purge at appropriate rate (25-30) by adjusting flow indicator (marked "1"). Adjust them slowly as they are quite sensitive.
7. Make note of when He cylinder was changed in log book.

Radiation Enclosure/Safety Interlock

The radiation safety interlock prevents accidental exposure to X-rays.

The shutter will not open if:

- the safety interlock mechanism is not switched on;
- the R-AXIS controller is not on.
- the doors to the enclosure are open;
- the doors are closed but the reset has not been hit (green light on);
- the shutter switch is not on EXT (external);

The shutter will close if:

- the doors are opened while the shutter is open

The HT cannot be turned on if:

- the shutter switch is open???
- the shutter is mechanically jammed open.

The HT will be killed if:

- the shutter is mechanically stopped from closing;
- the R-AXIS controller is switched off;
- the safety interlock mechanism is switched off.

Both safety interlock and R-AXIS controller have to be on before X-rays can be produced. The safety interlock mechanism must be left switched on at the 240VAC socket above the R-AXIS controller. Doors must be firmly closed. Reset safety interlock by pushing yellow button on box next to right-hand door - the green light should come on. (IF it doesn't, check that doors are properly closed). Shutter may now be opened.

To go into enclosure, check that shutter is closed and then simply open doors - green light will go out.

Osmic confocal max-flux optics

Users never have to touch these. However users are responsible for checking that helium is flowing through the optics. **Direct beam shots** are performed **only** by the X-ray manager following a filament change and alignment, and the value is hard-wired into the CrystalClear software. If users wish to use other processing packages, such as MOSFLM, the latest direct beam position can be found in the log book.

Microscope

Unlike the microscope on the R-axis IIc, the position of the microscope is easy to adjust. So the beam will be located at the center of the cross-wires.

Goniometer

1. To alter the height of the goniometer, unlock using an allen key inserted into the hole located below a groove in the top grip ring with the ϕ angle graduations marked above it. The height can then be adjusted by turning the lower grip ring. Retighten the allen screw.
2. The goniometer can be rotated by hand for centering the crystal by turning the silver knob on the black upright on the LHS of the ϕ axis, so that "FREE" points west. To lock turn clockwise so that "LOCK" points west. Remember to lock before starting data collection.

R-Axis IV++

Switching On

1. Check that the small silver and black Rietschle vacuum pump located on the floor behind the R-axis IV++ is running. If it is not, it is probably not plugged into the grey TEC transformer located on the floor behind the 60l liquid nitrogen dewar. Push the yellow plug into any of the transformer sockets.
2. Check LD switch on the back of the R-axis IV++ is set to "auto".
3. Turn on the blue controller, located on the floor at the back RHS of the generator, using the red toggle switch. The green light will illuminate.
4. Check the HV switch on the back of the R-axis IV++ is on. It will glow green if it is.
5. Check that the "pizza box" interface below the PC monitor is on (switch on RHS, and green light illuminated if power is on).
6. Reboot PC.
7. To shutdown simply switch off the blue controller box located on the floor at the back RHS of the generator using the red toggle switch. The green light will go out.

Changing the crystal to detector (XTD) distance

The crystal to detector distance can be varied from 70mm to 450mm. Loosen the silver screw on top of the block at the back LHS of the R-axis IV++. Use the black turn handle to move the R-axis IV++ back and forth. A digital readout box to the RHS of the R-axis IV++ gives a measure of the distance in mm. **The XTD distance is the only R-axis / generator parameter that is not read directly by the computer. It is therefore vital that this distance is correctly entered manually into the CrystalClear software during setup.** If the digital readout loses power, the readout will start flashing. Move the detector to its furthest position and press the * button on the box. This resets the XTD distance to its maximum value.

PC

Username	raxis4
Password	"password"
Processing disks	D: or E:

CrystalClear

To launch CrystalClear, click on icon (bottom LHS). Username is raxis4, and leave the password blank. CrystalClear controls all aspects of data collection and can fully process data using 3-D profiling for integration. Always use a new sample name for every new crystal. Do not use DEC incompatible filenames, in case you want to process on the main-frame. To optimise data collection for data processing with CrystalClear, a fine ϕ slicing strategy should be employed. The defaults work well in most cases, but the user must remember **to enter the XTD manually**. Follow the flow chart through on the LHS of the window to set up a data collection, including taking test images, indexing, and devising a data collection strategy. Following data collection the software can refine your cell, integrate and reduce your data, outputting graphs of completeness, Rmerge, I/σ etc. Most of this can be accomplished by simply altering options in boxes. The

only part of the software that is not intuitive regards making a mask for the excluded area of the backstop prior to integration. To make the mask first click on the toolbar button showing a circle. Click with the LH mouse button in the centre of the backstop circle and keeping your finger on the LH mouse button draw out to the edge of the circle and release. A circle will now be blocked out. To remove the area of the backstop support, click on the rectangle toolbar button. With the LH mouse button click at the 4 corners of the area to be excluded. A 4-sided area will now be blocked out. To write out the mask file click on the toolbar button to do this. Use the default mask name.

Backing up data

1. Get a special DAT tape from Don Akrigg or Mike Beck. Insert into the DAT drive on the PC.
2. Click on the START menu, scroll up to Programs, then HP Colorado Backup II, then click on the icon "HP Colorado Backup II".
3. Choose the option "Backup Wizard" and click "Next>".
4. Choose "Backup selected files, folders and drive" and click "Next>".
5. The Explorer style window shows drives and folders in the left pane and files in the right pane. Drives and folders are expanded by clicking on the + symbol, and the contents of the folder can be displayed by clicking on the folder icon. Select the files, folders or drives you wish to backup by clicking in the box beside it to enter a tick mark.
6. Click "Next>".
7. Choose either "All files" or "New and changed files" to perform either a full or incremental backup.
8. Click "Next>".
9. Make sure that "HP C5683A Backup Device" is selected as the backup device.
10. Click "Next>".
11. Toggle the comparison and compression options according to your preference.
12. Click "Next>".
13. Choose to do the backup now or later. The following details assume that an immediate backup has been chosen.
14. Click "Next>".
15. Give the backup job a name.
16. Click "Start" to commence the backup job.

Cryostream

Liquid nitrogen delivery.

1. The person collecting data on any Tuesday is responsible for getting the 200l liquid nitrogen dewar filled.
2. Liquid nitrogen is delivered every Tuesday between 10:00 and 13:00. **Make sure the dewar has been taken down prior to 10:00, Tuesday.**
3. Take the dewar down to level 4 of Biology in the goods lift just outside the level 8 door separating Astbury from Biology. **Never travel with liquid nitrogen in the lift.** Press the level 4 button before closing the lift doors. If the lift does not go down, then the doors are not shut properly.
4. Travel down to level 4 separately, and remove dewar remembering to close the lift doors. The dewar should be taken around the corner and left in the corridor by the loading bay doors.
5. Remember to pick the dewar up early afternoon. A delivery note will be lodged in the top of the dewar. Give this to the X-ray manager.

Cryostream operation.

1. Check dry air unit is running (power knob at top RH corner). Normally the dry air unit should be running continuously. If it has been switched off, it needs to run for 24 hours to dry down the columns before the cryostream is switched on.

2. Top up the liquid nitrogen dewar. This can be done whilst the cryostream is running. The dewar is large enough to run the cryostream for 48 hours, so if your data collection is more than 48 hours in length, you should temporarily suspend data collection, and then restart from where you left off after topping up the dewar. Wear the large blue cryogloves and glasses. Pull the white plastic bung further up the cryostream leg to allow access. To make sure ice does not get into the liquid nitrogen (which could block the flow in the cryostream leg), turn on the liquid nitrogen flow from the dewar, and direct it into a polystyrene box until the transfer pipe has cooled down enough that liquid nitrogen is flowing out of the end rather than gas. Switch off the flow, and put the pipe into the 60l dewar, making sure it goes all the way to the bottom. Switch back on until the liquid nitrogen begins to spit from the top of the dewar (it takes several minutes if empty). To make sure you have fully topped up the dewar, pull the pipe out until the end just catches beneath the neck. Turn the liquid nitrogen on only slightly until spitting again occurs. Shut off liquid nitrogen and remove transfer pipe. Push back down the white plastic bung into the top of the dewar.
3. Switch on the controller using the toggle switch at the back (lower RHS). It will go through a checklist and then allow you to program the unit. Press program and use the single up or down arrows on the RHS to select "RAMP". Press enter, and then use the arrows (double arrows alter in units of 10, single arrows in units of 1) to give a rate of 300 (press enter) and then a final temperature of 100K (double arrows alter in units of 10, single arrows in units of 0.1) followed by pressing enter. Select "HOLD" from the program list and press enter (the holding temperature will automatically be the final ramping temperature). Press start. The cryostream should take about 40 minutes to reach 100K.
4. When finished, use the up and down arrows to select "END" (you may have to press program first, if a black box with a program option is not visible below the current instruction). Press enter. With arrows select ramp speed to be 360 and final temperature to be 290K, pressing enter between each step. Press start, and the cryostream will ramp back up to 290K and then shut down. The start up again, the controller will need to be powered off and then back on using the power toggle switch at the back (lower RHS).
5. If small ice crystals get up the leg, the flow rate will drop and the cryostream will shut down. In this case the leg must be taken out, propped vertically and allowed to warm up fully and water to drain out. Leave for 2-3 hours to make sure all water is removed. The dry air (red) line can be removed from the head and the dry air blown up the leg to help dry it out. Replace back into the 60l liquid nitrogen dewar and restart.
6. The 60l dewar should never be left without some form of bung in the top, otherwise a huge plug of ice will form.
7. Periodically, if not in use, the 60l dewar should be dried out by when the nitrogen has boiled off, turning it upside down and letting any water drain out and the dewar fully dry.