

## R-AXIS IIC Hardware Documentation (User guide)

### Rigaku RU200 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 RH corner of the room should start to hum and the "line" light on the control panel at the back of the generator should come on. If not, the switch on the main distribution board may have tripped off (this happens fairly frequently). This is in the locked junction box by the window in the R-axis IV++ enclosure. Only Don Akrigg and the University electricians have the key. Make sure the isolator is switched off, try switch back on the appropriate fuse and try turning on the isolator behind the generator again. (You may have to do this several times).
2. Turn on the main power section at rear of the generator, near the floor. This illuminates the power LED on the vacuum controller sub-panel at the rear of the generator, and the power and ready LED's on the TMP drive unit sub-panel also at the rear of the generator.
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/cm<sup>2</sup>  
TUBE FLOW - goes out when water flow to tube below 6L/min  
TARGET FLOW - goes out when water flow to target below 8L/min.  
If either of the flow lights is out, adjust inline valves before flow meters (see “in-line water filter” section above).

4. The vacuum controller will normally be left in auto mode - on the vacuum controller sub-panel at the rear of the generator, the A/M switch should be in AUTO position and the DEGAS switch should be off (lower position). 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 (on the TMP drive unit sub-panel at the rear of the generator, the ready LED will go out and the accel LED will come on; when the TMP is at full speed, the accel LED will go out and run LED will come on - the TMP LED on the vacuum panel at the front of the generator will also be illuminated). About five minutes later the ion gauge will come on (the IG LED on the vacuum panel and on the vacuum controller sub-panel will both be illuminated). It will now be possible to take readings of the ion gauge using the multimeter connected to the vacuum controller sub-panel at the rear of the generator. Initial readings may be as high as 350mV. When the vacuum gets below  $10^{-4}$ Pa (approx. 150mV on the multimeter), the operated LED on the vacuum panel and the  $10^{-4}$ Pa LED on the vacuum controller sub-panel will both come on.
5. If the filament has been changed and the ion gauge has been exposed to the atmosphere, measured values from the IG may differ from actual values. It is thus necessary to degas the IG at this stage to drive out adsorption gas. Turn on the DEGAS switch (up is on) on the vacuum controller sub-panel at the rear of the generator. The DEGAS LED will come on. Leave on for five minutes ONLY. The IG will cease working during degassing and the multimeter will give a zero reading. Remember to turn DEGAS off after five minutes.
6. 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.
7. 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. (If the flow rate to the target was adjusted earlier, it may be necessary to reduce the flow using the inline valves. This is because resistance to flow decreases when the anode is rotating). The ion gauge reading will go up, so wait for it to come down again before proceeding further (reading below 100mV).
8. 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.
9. If the filament has been replaced by the X-ray manager, it should be aged according to the following protocol. The voltage and current can be taken up in 10kV/mA steps, allowing either 30 minutes (1 hour for the 20kV 10mA step) or sufficient time for the IG reading to fall below 100mV after each adjustment. If the filament is not new, power can be increased more quickly (10 minute gaps) but still requiring the IG to be below 100mV. This is to allow the filament to heat up slowly after increase.

kV	mA	kV	mA
20	10	50	50
30	10	50	60
30	20	50	70
30	30	50	80
40	30	50	90
40	40	50	100
44	44		

The tube voltage and tube current can be adjusted using the UP/DN buttons on the X-ray panel. This raises/lowers the settings in 2kV/mA steps, and the settings are digitally displayed by the kV and mA indicators on the X-ray panel. The filament current should not rise above 1A as shown by the meter on the front of the generator. The maximum power of 50kV, 100mA corresponds to 5kW.

**N.B.** Make sure that there is helium flowing into the mirror box before opening the shutter (see mirrors).

## Shutdown

1. Lower tube voltage and current to the minimum values (20kV and 10mA). This is done by the reverse of powering up (above), but can be done relatively quickly as the filament does not need to cool down slowly.
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. If the A/M switch on the vacuum controller sub-panel is set to AUTO, simply press SYSTEM STOP on the vacuum panel to stop system exhaust. The vacuum controller sequentially stops the TMP and then the rotary pump. As the TMP is slowing, the ACCEL LED on the TMP unit drive sub-panel will start blinking. As the rotating speed becomes even lower, the ACCEL LED blinks at longer intervals. The READY LED will become illuminated when the TMP has stopped. Continue with cooling for at least 10 minutes to allow the TMP to cool.
4. Turn off the power switches ELB1 and NFB1 on the power section at the rear of the generator.
5. Turn off the isolator for the X-ray generator 3-phase power supply.

## Helium Supply

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

### Changing Helium Cylinder

1. Turn off HE beam path flow control (HBPF) for both the mirror box and He bellows (if being used).
2. Close valve on He cylinder.
3. Replace cylinder with new one from stores (storeman has key).
4. Open valve on cylinder.
5. Adjust He regulators, if necessary, until they deliver flow rate (keep below 1.0 bar).
6. Remove small hose from collimator and yellow cap from He bellows.
7. Set both HBPF's to flood for about 5 minutes. Flow indicators (marked "H") on HBPF's should not be too high.
8. Set HBPF's to slow purge at appropriate rates (25-30) by adjusting flow indicator (marked "I"). Adjust them slowly as they are quite sensitive.
9. Replace tube on collimator and yellow cap on He bellows
10. Make note of when He cylinder was changed in log book.

### He Bellows

Helium bellows reduce adsorption of X-rays by the air when the xtd distance is about 150mm or greater.

1. Set the R-AXIS at the correct distance.
2. Remove the protective pieces of cardboard from the He bellows. Also, remove the yellow cap so that the bellows can be expanded and collapsed without bursting the Mylar windows.
3. Without touching the Mylar windows, carefully attach the legs to the outside rails of the R-AXIS sled using the Allen screws. Don't do the screws up too tightly, the position will probably have to be adjusted. Have the smaller Mylar window facing towards the X-rays.
4. Turn off the PMT HV at the rear of the R-AXIS - a red light should go off.
5. Above the IP aperture, carefully remove 2 screws from a small stainless steel disc and keep these screws safe.

6. Above and below the IP aperture there are rows of 4 screws. Remove the outer two from the upper four, and the inner two from the bottom four, and put them aside.
7. Using the longer Allen screws kept in the Petri dish marked "Screws For He Bellows", loosely attach the two black metal brackets to the front of the R-AXIS, in the positions where the screws have just been removed - the longer bracket at the top with the cut-away edge facing down/towards the IP, the shorter bracket at the bottom with the cut-away edge facing up/towards the IP.
8. Position the frame of the larger Mylar window so that it hooks behind the cut-away edges of the metal brackets, and so that it is centrally located over the IP aperture. Tighten the screws on the brackets so that the frame is held securely against the R-AXIS.
9. Move the legs of the He bellows as far forward as they will go, without the frame touching the  $\phi$  goniometer, and tighten the screws.
10. Attach the tube from the HBPFC to the top hole on the front frame of the bellows. (Helium is lighter than air and so air inside the bellows is displaced downwards by the incoming helium). Set the HBPFC to flood for approx. 5 minutes.
11. Turn the HBPFC to slow purge (flow rate of about 5 units). Attach the yellow plastic cap to the bellows. Attach the oil bubble chamber to the bottom hole of the bellows. Adjust the flow until about one bubble chamber to the bottom hole of the bellows. Adjust the flow until about one bubble every 30 seconds.
12. Turn on the PMT HV.

Note : If you leave the bellows attached but change the XTD distance, remember to remove the yellow plastic cap.

### **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 but do not have to be locked. (To close the left door, pull out on key and slide door gently closed). Reset safety interlock by pushing small black 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.

## Mirrors

The only part of the mirror box that will need to be adjusted by users is the thickness of filter on the filter wheel inside the mirror box for direct beam shots. This is controlled by the Focusing Mirror Optics Controls. Press the red button to turn this unit on (the button illuminates). The selector should be on position "4, filter". Use the "forward, reverse" button to rotate the filter wheel. The thickness of Ni filter is indicated on the wheel. Position the bottom of the circle of Ni is in line with the metal dot. For data collection a 0.0006" Ni filter is appropriate, and for direct beam shots the filter should be used. **To take a direct beam shot** move the 0.006" Ni filter into place. Remove the backstop and take a 2 second exposure. Replace the backstop and 0.0006" Ni filter.

## Microscope

Following a filament change the vertical position of the beam relative to the microscope changes. Adjusting the height of the microscope accurately is a non-trivial exercise for the X-ray manager. So the position of the beam along the central vertical cross-wire may vary, and **may not be on the center of the cross-wires**. **Users must check the logged beam position relative to the microscope cross-wires. This will be located in the log book as a graphical drawing. Look for the latest entry of this type in the log book.** The alignment horizontally of the microscope is much easier, so the optical centre rotation for the crystal will be along the vertical axis of the cross-wires.

## Goniometer

1. The height of the goniometer can be altered by unlocking the second allen nut down on the base with the goniometer key, and turning the grip ring below the nut. Lock nut when the adjustment is finished.
2. The goniometer can be rotated by hand for centering the crystal by unlocking the top allen nut on the base with the goniometer key. Remember to lock before starting data collection.

## R-Axis IIc

### Switching On

1. Turn on the 240VAC to 115VAC step down transformer at the mains socket above the controller. This feeds the controller and the IP readout section.
2. Turn on the controller at the switch near the floor (labelled "power"). The switch will be illuminated.
3. Turn on the power switch (labelled "power") of the Linear Motor Power Supply. It will become illuminated. Check that the "drive" switch is on, and that the lamp is illuminated.
4. Check that the "power" and "HV" switches on the HV/ADC unit are both on. The lamps may not be illuminated at this stage. This supplies HV to the photomultiplier tube (PMT) and also digitises the output signal.
5. Turn on the power switch of the IP readout section. This is located at the back, on the right-hand side. Make sure that the side panels of the readout section are closed. Turn on the HV switch, also located at the back. A lamp will become illuminated. This supplies HV to the PMT. **Do not turn on the HV if the side panels are open, as it will blow the PMT.**
6. To shutdown reverse sequence.

If the AlphaStation BMBVS7 is rebooted, it occasionally loses sight of the R-AXIS and complains "device is not in configuration or not available". The remedy to this is to get Don to shut down VS7 (the command when logged onto VS7 as SYSTEM is @sys\$system:shutdown). Reset (see next section) the R-AXIS controller while VS7 is down (it is not necessary to turn off the X-rays to reset the controller - turning off the controller kills the X-rays because of the safety interlock, whilst resetting does not). At system level, type "show device" and look for "a/7/0" which is the ID for the R-AXIS device. Type "b" return to reboot VS7. You must then reset the controller again, otherwise you'll continue to get "timed out" errors by pressing the button until red LED turns to green LED.

On a DECwindow type "show device gka700:". This device (the R-AXIS controller) should be online. The ultimate test though is whether you can initialise the R-AXIS.

### Resetting the controller

The right-hand circuit board in the controller marked CPUM is the mother board (6<sup>th</sup> board). The SCSI cable from VS7 is plugged into it, as well as a serial line. Between the two cable connections on the edge of the board is a small button. Behind that button (about 10cm further back on CPUM) are 3 LED's. Normally the green LED will be illuminated. To reset the controller, depress the button on CPUM so that the green LED goes out, and a yellow LED comes on. Keep the button depressed until the yellow LED goes out and a red LED comes on (a few seconds later), and then release the button. The red LED will go out and the green LED will come back on.

### Changing the crystal to detector (XTD) distance

The crystal to detector distance can be varied from 65mm to 200mm by the means of a screw mechanism attached to a metal knob at the rear of the IP readout section. This must be adjusted by hand. It is best not to change the XTD by simply pushing the readout section, as this may move the position of the sled and thus the screw up alignment. It may be necessary to oil the track on which the readout section slides when its movement begins to stiffen. The distance can be read fairly accurately using the vernier at the front of the R-Axis sled.

The beam centre should change very little (maybe by a couple of pixels in the horizontal and vertical directions) when changing the XTD. (The fact that the beam centre does not move when varying the XTD also confirms that the IP is perpendicular to the X-ray beam).

### AlphaStation BMBVS7

Username	raxis
Password	"password"
Default directory	dsk\$36:[raxis]
Processing directory	dsk\$36:[raxis.dat.blah-blah]
IP directory	dsk\$37:[raxis.dat]

The default directory when you first log in is dsk\$36:[raxis]. Scratch images ipdat1.dat (105µm pixel) and ipdata2.dat (210µm pixel) are stored in dsk\$36:[raxis.data]. When the IP's are read in manual, the images will be read to these files. Most users will not need to write images this way, as this is not useful for data collection purposes. **But do not delete these files, as the program needs to find them present.**

Create sub-directories in dsk\$36:[raxis.data] for data processing. You will need files crystal.dat and scale.dat in this created sub-directory for processing.

**Images must be written to dsk\$37:[raxis.data].** This is an external disc drive and should have sufficient space, whereas dsk\$36:[raxis.data] is an internal disc and will quickly fill up, crashing the computer. Check the amount of free disc space on dsk\$37: before use by "show device dsk\$37:". Each image is 15,208 blocks, so judge whether you have enough space on dsk\$37 for data collection. Still images will be called file\_name.stl and oscillation images will be called file\_name.osc. **Back up your images as soon as possible.**

The program to control the R-AXIS can be started by typing "Start". This also runs the graphics and data processing software.

Hardware control software can be run by typing "Adjust", but most users will not need this.

## 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 only large enough to run the cryostream for 24 hours, so if your data collection is more than 24 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. Remove the cotton wool bung. 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 25l 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. Replace cotton wool bung.
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, press program and select "END". 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 25l liquid nitrogen dewar and restart.
6. Periodically, if not in use, the 25l 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.