

**X-ray Laboratory at Glasgow  
Biomedical Research Centre,  
Level 2, Room B219**

**Manual**

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**Approved by Neil Isaacs**

## X-ray Laboratory at GBRC, room B219



1. Rigaku X-ray generator MicroMax-007, micro-focus beam, 800-Watts (40kV/20mA) maximum power.
2. Rigaku Osmic confocal optics mirrors serving as a monochromator and collimator of the X-ray beam.
3. Helium cylinder and controls for helium gas running through Osmic mirrors.
4. Water chiller cooling the closed-circuit water, which absorbs heat from the generator anode.
5. MarResearch image-plate detector Mar345 located on the detector table (dtb).
6. Mar345dtb-controller with ion chamber IC<sub>1</sub>/IC<sub>2</sub>-readings and chi-angle control button (among others).
7. Oxford Cryosystems AD41 Dry Air Unit: produces dry air for the Cryostream coldhead.
8. Oxford Cryosystems 600 Series Cryostream Cooler controller.
9. Cryostream coldhead: produces stream of nitrogen gas to flash-freeze the crystals to 100K.
10. 60-liter open Dewar with liquid nitrogen for the Cryostream coldhead.
11. 160-liter pressurised Dewar on wheels for the transport of liquid nitrogen from the fill-up area.
12. 20-liter egg for small amounts of liquid nitrogen to be used for freezing tasks.
13. Frozen crystals storage Dewar (Taylor-Wharton LS-750) with liquid nitrogen level control and alarm.
14. Microscope (Olympus SZ40) for examining crystals and looping them out for diffraction experiments.
15. Computer (outside of glass enclosure) dedicated to run Mar345dtb (beam alignment and data collection).
16. Door in the glass enclosure (access controlled by the safety switches to the right – not shown here).

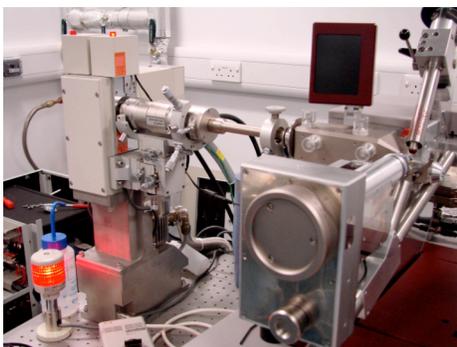
# I. Entering the glass enclosure = Entering Controlled Radiation Area



Safety installations of the X-ray System glass enclosure make it impossible to enter the area when the X-ray generator shutter is open. The shutter is being automatically closed when someone opens the door preventing anybody being exposed to harmful X-ray radiation. When the user has finished his/her work inside the enclosure he/she needs to leave the area and close the door tightly. Only then the X-ray generator shutter can be opened with the use of the switchboard located on the wall to the right of the door. [Read the info on the system of work attached to the door to refresh your memory.](#)

## To open the X-ray generator shutter one has to:

1. Close firmly the door,
2. Press the yellow RESET button – this should light on the green light RESET ON located above the yellow button. In case that does not happen, correct the door position and try again.
3. Switch the SHUTTER switch (left bottom corner) from OFF to ON position. In case it was already in ON position, switch it OFF first, then ON.
4. When the shutter gets opened, small red bulb on the top of the X-ray tube column lights up and the readings of the MAR detector ion chambers start showing values significantly higher than  $\sim 10.0/\sim 10.0$ . The large red lamp next to the X-ray tube column is on all the time when the generator generates X-rays, regardless of the state of the shutter.



Shutter closed



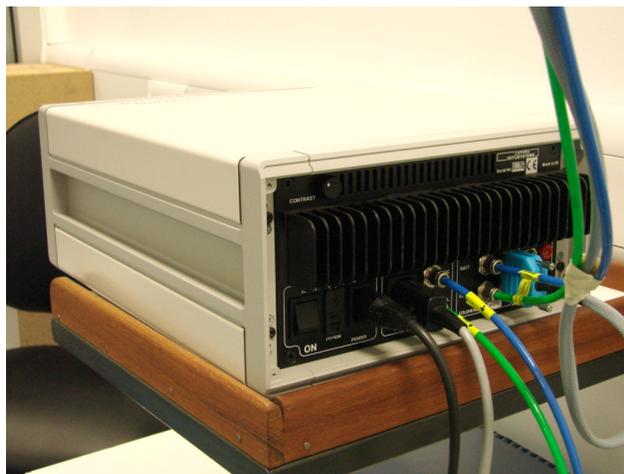
Shutter open

## II. Preliminary Operations before the X-ray Diffraction Experiments

1. Check that the Helium flow is open in a SLOW PURGE position (move switch to SLOW PURGE position if it is in OFF position) and that it is set to 20-30 ml/min rate, the latter can be adjusted with the right-hand (SLOW PURGE) vertical flow meter. Do not use FLOOD position/meter.



2. Switch the D41 Dry Air Unit on (behind the generator, shown below, left photo) before starting the Oxford Cryostream. **If forgotten, the nozzle of the Cryostream coldhead next to crystal position will freeze up and that will require stopping the Cryostream to warm the tube and to remove the ice.**



3. Check the contents of the 60-L Dewar (standing behind the generator) connected to the Cryostream coldhead by moving it gently. If it feels like quite empty go to point 8 for fill-up instructions. Otherwise start the Cryostream by switching on the POWER switch at the back of the controller (at the bottom left corner, see right photo above). The LCD window at the front of the controller should then display the start-up procedure checking all the essential components of the system (see top-left photo on the next page).



- When that is finished, press PROGRAM button (this button sometimes does not work first time, press it again). The screen should then change to one shown in the right photo above. Program the **ramping-down** process (lowering the temperature - in Kelvins - of the gaseous nitrogen coming out of the coldhead) with the use of up & down buttons (single 'arrow' gives smaller change, double 'arrow' bigger) and the ENTER button. If possible, follow the scheme shown below (left photo). The 360 K/hr rate is the fastest possible while 120 K/hr is the slowest. It is advisable to use the slow ramping at the end. In the third line the RAMP can be changed to HOLD but the holding at the final temperature will also be done without programmed HOLD. To start the ramping process press button START. After that you will hear the gas pump gurgling and the gas flowmeter on the left indicating flow of 4 litres/min, reaching 5 litres/min in few minutes (see the top level of the black indicator).



- Switch the screen of controller to the one shown in the right-hand photo above by pressing the SCREEN button. This screen gives you the status of the Cryostream, reporting the actual rate of ramping, remaining time (in bracket) and the gas stream temperature (in K). Additionally, three other parameters are listed, the gas temperature Error, the temperature of the evaporator heater EvapT, and the gas heater Heat %. These three parameters are very useful in diagnosing the possible causes of the Cryostream failures. If needed more help can be found in the Operation & Instruction Guide of the Cooler (on the table below the controller).

6. When the ramping process is finished you want to see the screen like this:



7. **Program the ramping-up process at the end of your work.** Choose one step ramping at the highest rate of 360 K/hr leading to a room temperature (290 K will do). When that is finished press button STOP, then switch off the POWER button at the back of controller. Finally, **switch the Dry Air unit off as well.**
8. Refilling of the 60-L Dewar (left photo below) from the transport 160-L Dewar (Wessington Cryogenics vessel, right photo below) is done with the 3 m hose attached to the 160-L vessel. To do that, place the end of this hose in the 60-L vessel and slowly open the valve next to the point where the hose is attached. Stop the transfer by closing this valve when the flow of LN<sub>2</sub> out of 60-L vessel is observed. The gauge mounted at the top of the 160-L vessel indicates the level of its contents. Refilling of the 160-L vessel is normally done by the BOC person when he delivers liquid nitrogen for the GBRC to fill-up the two parking-located big Dewars. That is being usually done twice a week (Mon or Tue, and Thru, late in the morning) so 160-L vessel has to be taken to that area in the parking using the goods elevator. **Refer to our Local Rules for Handling Liquid Nitrogen for more details on LN<sub>2</sub> transport and safety. During all the above operations use the safety goggles/face screen, gloves and the portable oxygen level monitors!**

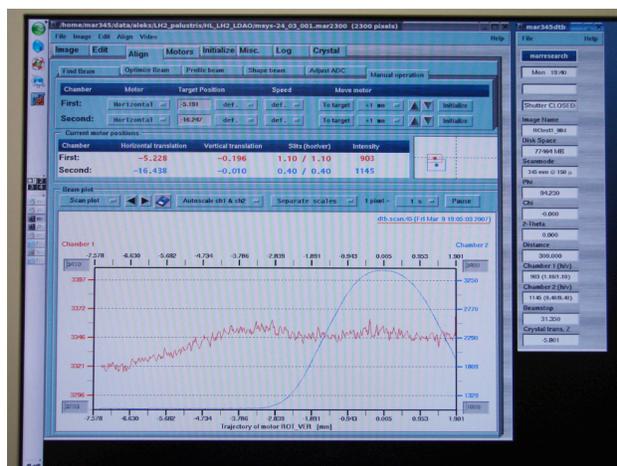
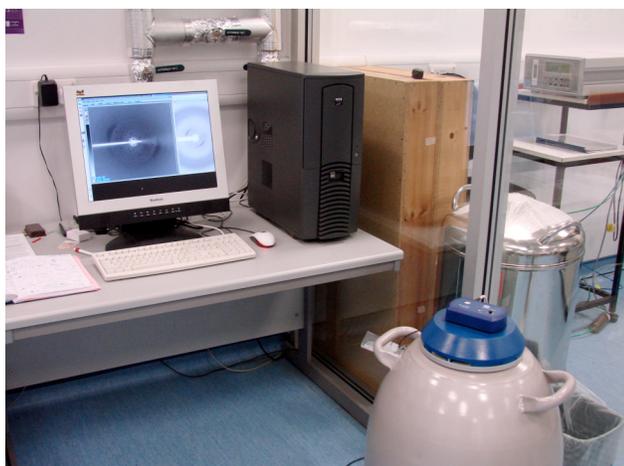


### III. X-ray Diffraction Experiments with the use of the Mar345dtb detector located on the MicroMax-007 generator

#### A. Normal Operations:

Normally, the X-ray generator is running at full power (40kV/20mA) and the beam is aligned so the user just needs to mount his/her crystal on the goniostat, centre it, leave the enclosure area and open the generator shutter with the use of the switches located next to the door (see chapter I above and the pictures therein). At that point one can right away see what the intensity of the beam is. It is measured by the ion chambers IC<sub>1</sub> (top panel of the display) and IC<sub>2</sub> (bottom panel) of the detector controller plate.

These values could be somewhere between 160.00/120.00 and 120.00/90.00 depending on the age of the anode and the condition of the filament. If the IC<sub>1</sub>/ IC<sub>2</sub> values are significantly lower than the above values but significantly higher than the ‘no-beam’ ~10.00/~10.00 base values, user can try to realign the beam by using Optimize Beam procedure which can be started from ALIGN tab of the *mar345dtb* software on the detector dedicated PC outside of the glass enclosure (left photo below).



If program *mar345dtb* is not running already, or if you want to open it in your own folder (in which case you first need to close the running program), open the terminal, go to folder [/home/mar345/data/'user\\_name'](#) and start the program *mar345dtb* with the command ‘GO’ or ‘*mar345dtb*’.

Two windows should open, main one containing tabs: **Image, Edit, Align, Motors, Crystal**, etc., and second, narrow one, showing the status of the detector, image name, present phi, chi and 2-theta angles, detector-to-crystal distance, beamstop position, crystal Z-translation (position of the crystal along the rotation/goniometer axis) and the ion chamber readings.

#### The most important tabs for the user and their main functions (mandatory functions in blue):

##### **Edit**

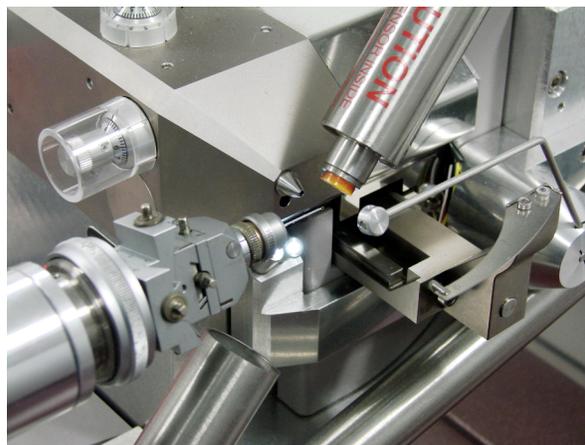
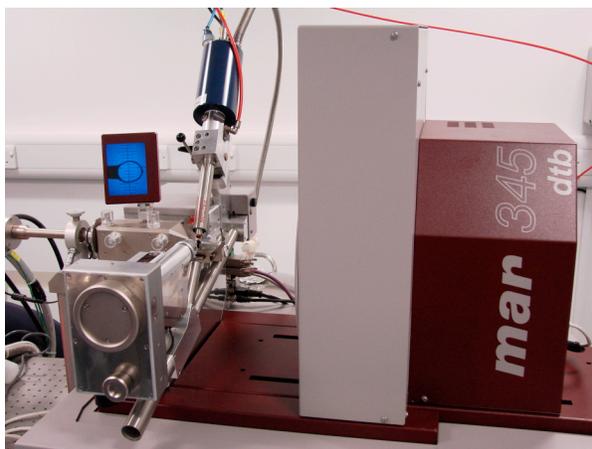
- In this tab diffraction experiment is defined and started
- Choose the folder for the new image(s) and [the image\(s\) name\(s\)](#).
- Select the scanning mode between pixelsize of 0.15 mm (for diffraction testing) and 0.10 mm (possibly for data collection, longer scanning), or between the full or partial image, and the image format (use *mar345*).
- [Choose the exposure time \(default is 60 sec\)](#); do not use ‘exposure dose’ option.

- Choose the number of images per block (keep no. of PHI-blocks as 1) and the first image number.
- Choose the number of oscillations per image, 5 min or less exposure per 1 oscillation recommended.
- Choose the image width,  $\Delta\varphi$ , per image (default is  $1.0^\circ$ );  $\Delta\varphi$  should be chosen depending on the longest cell dimension and on the image resolution, but it is not critical for crystal testing.
- Choose the  $\varphi$  increment which will be applied between images. Use  $0.0^\circ$  for a continuous data collection and  $90.0^\circ$ , for instance, for two snapshots at  $\varphi$  of  $0^\circ$  and  $90^\circ$ .
- Goniometer Settings, like 2-theta, phi ( $\varphi$ ) and chi angles, and the detector distance, can be changed from this tab/window, of which specially useful is the change of  $\varphi$  angle (for instance, to set it conveniently at  $0^\circ$  at the start of centering the crystal, or to any other angle of your choice to start data collection from), and the detector-to-crystal distance – the latter change will produce the new resolution limits for the image and these will be listed within the window.
- Collimator and Beam Settings can also be changed from this tab/window, most important of these will be the beamstop position: keep it at maximum (45.15 mm) to minimize the beamstop shadow and to maximize the space near the crystal/loop position for the comfortable loop mounting, however, **this distance needs to be shorter if the detector has to be positioned at 100 mm distance or closer. User will be prompted to check if the requested change(s) of the beamstop and/or detector position(s) are safe and ultimately will be warned about the possible collision.** At the very close detector positions (below 100 mm) there could also be an additional shadow produced by the beamstop holder so the beamstop may need to be moved as close to the crystal as possible. **Do not however use the absolute minimum of 5 mm in which case beamstop is about to hit the crystal!**
- **For a comfortable mounting of the crystal loop on the goniostat move the detector to at least 250 mm distance ( $2.6\text{\AA}$  at the edge of the image) and move the beamstop to its maximum distance.**
- From the Options at the bottom of the window use ‘Erase detector before starting set’ for your first image of the day and after each case of stopping image exposure with the use of button ‘Stop NOW’.
- Pressing button GO at the bottom of the window starts data collection. This will open another window titled Data Collection Parameters with another button GO. If all parameters are OK start collecting by pressing GO. You can stop data collection anytime by using one of three buttons: ‘Stop NOW’, ‘Stop after IMAGE’ or ‘Stop after SET’. Window ‘Data Collection Parameters’, if closed, can always be open from menu ‘Image’ selecting ‘Progress’.

## Crystal

- Displays a mounted crystal on the computer screen at higher magnification than is available on the small screen above the detector collimator block.
- Press the button ‘Sample’ to brighten the crystal view.
- Press the buttons ‘Background’ and ‘Show paddle’ to improve the crystal brightness further. The paddle reflects the ‘Background’ light towards the crystal. **It is a very useful feature but it is better to ‘Hide paddle’ for the crystal mounting to have an easier and safer access. The paddle is automatically removed for the X-ray exposure.**
- ‘Video’ menu allows to zoom the crystal/loop picture and to save it with the command ‘Save Photo’. The JPG photo with the name beginning ‘dtb-’ and containing the time of saving will be saved in the folder in which the program ‘mar345dtb’ has been opened.
- **X and Y coordinates of the crystal cannot be changed from this tab/window - user has to change them by adjusting goniometer-head translations with the use of the screw.** It has to be remembered that the viewing camera is located under the beam collimator at a small angle to the X-ray beam so it is

‘seeing’ the crystal almost the same way as the beam does; due to that the ‘zero’  $\varphi$  angle puts the goniometer-head flat parts - translations - at the same small angle to the horizontal level.



- Z-translations of the crystal (along the rotation axis) can be performed from this window using ‘+/-’ buttons.
- Rotation of the crystal by the specific angle of  $\pm 90^\circ/180^\circ/270^\circ/360^\circ$ , and a rotation by an arbitrary angle  $\varphi$  can also be requested.

### Image

- Displays automatically newly detected image; extra features can be requested via menu ‘Image’.
- Other existing images can also be opened in this tab/window using menu ‘Image/Open’.
- Left and right arrow buttons allow moving between images with the same name.
- ‘+’ and ‘-’ buttons zoom in and out the image, right mouse button can choose the specific rectangular area for magnification. Zoom in further to display pixel counts; pixels of overloaded reflections will be shown in green with the count number ‘999999’.
- Middle mouse click within the image area gives the approximate value of resolution.
- Left mouse button can be dragged through the line of spots belonging to the same ‘lune’ to measure the related period of the crystal lattice. Within the newly open window with the peaks, mark the centre of the most left peak using left mouse, the most right peak with the right mouse, and count the number of peaks from left to right. Write that number in the ‘No. of peaks’ space and press Return to obtain the period along this line of peaks.

### Align

- Of several functions of this tab/window user is invited to use the ‘Optimize Beam’ function only. It allows optimization of the detector position relative to the crystal position automatically. Ion chamber IC<sub>2</sub> reading should indicate improvement after this procedure if the detector was misaligned. **It is critical that the shutter is open before starting beam optimization. Lack of the beam may cause the serious detector misalignment, which could be not recoverable even if procedure is started again with the beam. For this reason it is always worth to note the Horizontal and Vertical translations for both chambers and the slits dimensions prior to running ‘Optimize Beam’ process. They can be read from the ‘Optimize Beam’ window.**
- At present, these values are: IC1: Horizontal transl. -5.291, Vertical transl. -0.186, slits 1.10/1.10  
IC2: Horizontal transl. -16.522, Vertical transl. 0.389, slits 0.40/0.40

In case of complete loss of the beam, do not use 'Find Beam' procedure; use 'Manual operation' instead to insert the values shown above, or any other values which were observed prior to the misaligning process if significantly different. [It is however recommended that only an experienced user perform this procedure.](#)

## Motors

- This tab is used to change the Goniometer Settings, and the Collimator and Beam Settings, the same way as they are being changed in **Edit** (see the earlier description). The Z-translation of the crystal can also be changed from here as well as from the **Crystal** tab.

Tabs **Initialize** and **Misc.** should not be needed in the regular work, and only an experienced user should attempt using these tabs. **In particular initialisation of the Z-translation should not be done when crystal loop is on the goniometer-head and the Cryostream coldhead is in its normal low position. Present version of the *mar345dtb* program unfortunately drives the goniometer-head to the 'FAR end' position when Z-translation is being initialized and that leads to a collision with the collimator and/or the coldhead.** This hopefully will be corrected in the new version of the program. [At present, in case of the error related to Z-translation try to move the Z-translation back by hand but do not initialize it, and better consult Aleks if possible.](#)

The tab **Log** reports the main changes in the detector status like changes in motor positions, etc.

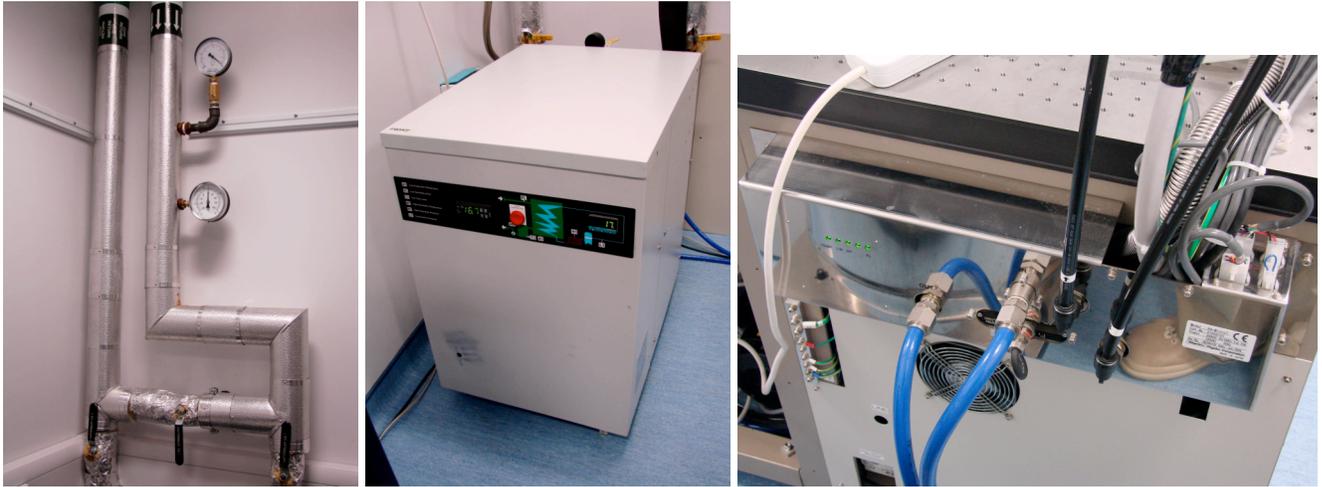
## **B. Abnormal Situations: Restarting the generator from the ALARM state.**

There are several possible situations, which will cause that the X-rays will be shut down and alarm will sound. The generator will never recover itself from such events and manual user intervention will be required. There are following main causes of an alarm:

1. Electric power-break in the building (rare). It will shut down X-rays and most likely will also stop the Flowcool chiller which is cooling the water in the anode circuit of the generator (connected to it *via* two blue hoses), due to external chiller (on the GBRC building roof) shutdown. This situation will require restarting Flowcool chiller after power is restored and then restarting X-rays. Both procedures are described in the next paragraph.
2. Failure of the Flowcool chiller itself, or of the chiller on the roof, other than the power failure. X-rays will be shut down with the alarm and one of the error messages: '**COOLING WATER FLOW ALARM!**' or '**COOLING WATER TEMPERATURE ALARM!**', due to insufficient water flow through the anode or too high temperature of that water, accordingly. This will require correcting or repairing the chiller and then restarting X-rays. See procedures in the next paragraph.
3. Filament burnout or in the process of breaking (happens once per three months on average). X-rays are off with the following error: '**FILAMENT CURRENT LIMIT ALARM**' displayed on the generator panel. One could restart X-rays again but if the situation repeats itself, the filament has to be replaced with the new one. Procedures for the replacement of the filament and for the subsequent filament aging process are described in the Rigaku '**RA-Micro7 Horizontal RotaFlex Instruction Manual**', however, these operations can only be performed by an experienced user or by the Rigaku personnel.
4. Vacuum system failure (very rare) due to problems with the turbomolecular pump (TMP) or with the front rotary vacuum pump. X-ray tube vacuum is compromised and X-rays are off with the following error: '**VACUUM ALARM**' or '**TMP OVERLOAD ALARM**' displayed on the generator panel. The source of the alarm has to be investigated and possible repair done by Rigaku personnel. The level of vacuum required for starting X-rays is  $10^{-4}$  Pa (equivalent to ..., both values shown on the generator panel when...)
5. There are several other reasons for alarm error messages, which are described on page 12 of the Rigaku Manual mentioned in point 3 above. Again, the source of the alarm has to be investigated and possible repair done by Rigaku personnel.
6. The **EMERGENCY** red button has been pressed on, intentionally or by accident, which shuts the X-rays off instantly. This will require removing the cause of the alarm, resetting the alarm by pressing the red button and turning it clockwise, then restarting X-rays using procedure in the next paragraph.

### **Flowcool chiller restart:**

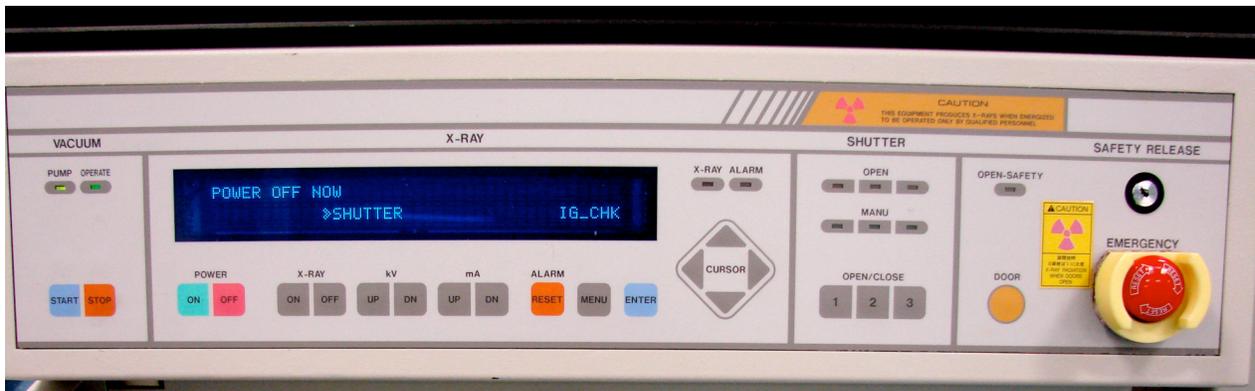
- Check if there is a pressure in the external water system (usually about 4 bar) and an appropriate temperature (supposed to be about 10°C) shown by the attached gauges (see left photo below). If there is a pressure but temperature is much higher, open the pipe bypass for few minutes; lack of either or both has to be reported to Graeme Tobasnick or to janitors.
- Shut off the power to the Flowcool chiller (middle photo below) on the wall.
- Reset the RESET button of the chiller through the hole drilled in the chiller's front panel (at the bottom left corner) using a pencil.
- Turn the power back on; in few seconds you should hear compressor starting and see front panel LCDs displaying present temperature of water. The chiller is set to hold temperature of 17°C in the anode water circuit and that ( $\pm 1^\circ\text{C}$ ) should also be restored in few minutes of compressor work.



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- If the chiller is working fine check the status of the most-right light (designated FL for ‘flow’, see the right photo above) of the row of five lights at the back of the generator, to the left from the blue hoses. If the chiller is on and the chiller’s pump in the anode circuit is running properly, that light should be on. This light will be on regardless of the actual water temperature. In the normal situation all five lights are on.
- If the chiller shows an error like **H1** while starting or fails with that error later on but at the same time it is known that the external water supply was not interrupted, or alternatively, the chiller does not hold the temperature 17°C (±1) (if the water in the anode circuit reaches high temperature, about 25-27°C, it will trip the X-rays automatically), call Peter Collins from MarResearch, tel. 01580-882051, or Industrial Cooling Services (ICS, ask for Caroline or C.J.), tel. 01425-625855, for help.

### X-rays restart:

- Reset the alarm with the ALARM RESET orange button (in the middle of the photo below) or if ineffective use the red button POWER OFF to the left. If the EMERGENCY red button (at the right end of the panel) has been activated, it has to be reset by simultaneously pressing it and rotating clockwise.



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- Press the POWER ON green button and wait a moment; the display should come up with the message: ‘POWER ON NOW’ and then ‘READY NOW’.
- Press the X-RAY ON green button; this should:
  - light up the large red lamp marked X-RAY standing on the generator table,
  - raise and display the tube voltage of 20 kV and the filament current of 10 mA, these are the minimum settings,

- display the message: ‘X-RAY ON NOW’ (see left photo below).



- Raise the tube voltage and filament current to maximum allowed values of 40 kV and 20 mA using appropriate grey buttons UP and DN (down). First set the voltage by stepwise pressing (not continuously) button kV UP. When 40 kV is reached message: ‘50% power’ should be displayed; if higher voltage was set by mistake, use button kV DN to reduce it. Then set the filament current, again in steps, up to 20 mA, which at the end should produce a message: ‘100% power’.
- Leave the generator running with X-rays on for 90 minutes to stabilise the beam focus position. **Do not try to optimize the beam by running the Optimize Beam procedure from the ALIGN software, nor by adjusting the mirror settings before the end of this 90-min period. Adjusting the mirrors settings is recommended to be done by an experienced user only.**
- After the 90-min stabilisation period close the glass enclosure door from outside, open the shutter and check the ion chamber readings. If they are reasonably high – as explained in the earlier chapter IIIA – do nothing or run the Optimize Beam procedure from the ALIGN tab of the mar345dtb software.

**Generator restart:**

If for any reason generator power was completely off, for instance for filament replacement, use the following steps:

- Turn the power on by using the ON button in the Main Power section located behind the door below the generator control panel (see photo below).



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- Press the blue button START from the VACUUM section (extreme left) of the control panel to start pumping vacuum. The left of the two lamps in this section labelled PUMP should start blinking green for about 20 seconds, after which time it should turn to steady bright green light. At that point the second lamp labelled OPERATE should start blinking and eventually turn to steady light (darker green) when certain required level of vacuum has been reached.
- Leave the generator running for a while and observe the level of vacuum. This can be done by pressing MENU grey button and from MENU display selecting option IG\_CHK (check ion gauge vacuum). This will open another display listing vacuum level in mV and in mPa (see photo below). Vacuum level of 200 mV or better (lower number) is sufficient for starting X-rays. The picture below shows the typical vacuum when the X-rays are set at full power and run for a while.



### **Mar345dtb-detector restart:**

If the detector has been switched off or it has to be restarted, switch on two separate green power switches, one on the back of the image plate and one on the mar-dtb below the cryosystem coldhead. In that case the mar345dtb software on the dedicated computer outside of a glass enclosure has to be closed and open again.

