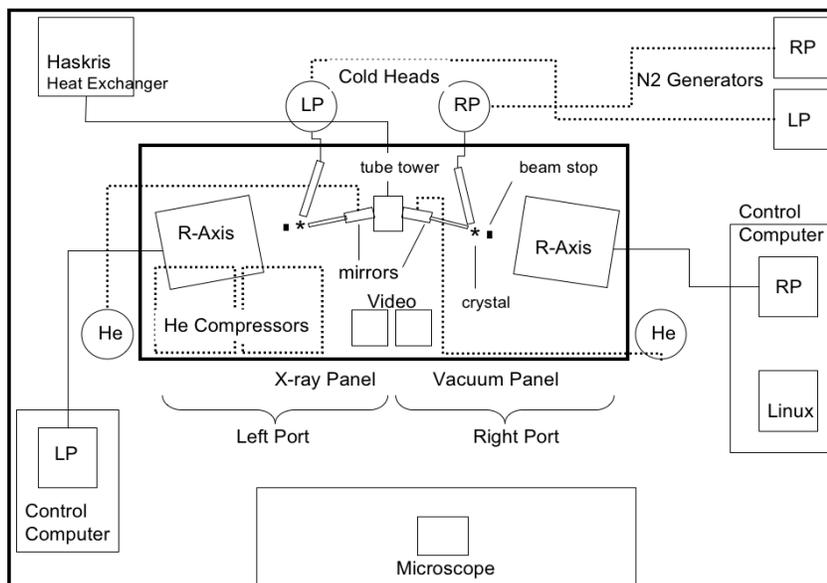


MOLBIOCH 840
Overview of X-ray Lab Equipment
Chuck Bell

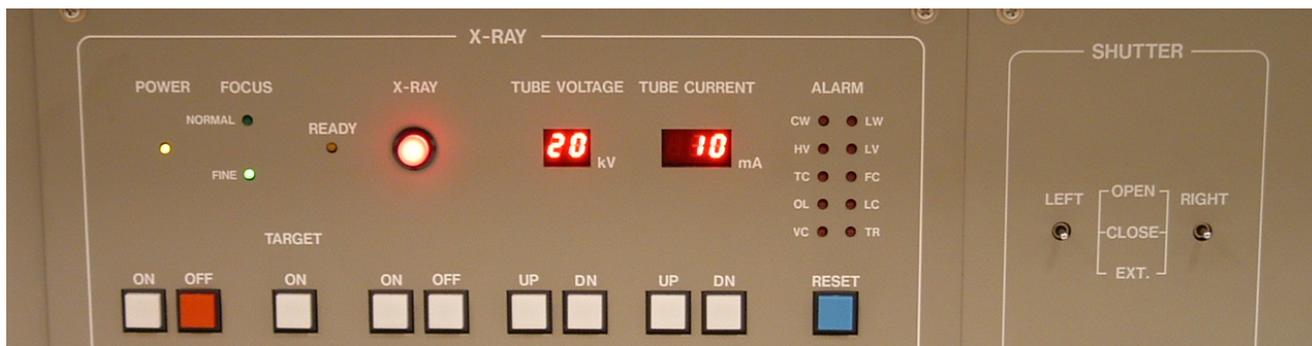
Equipment overview: the equipment in the x-ray lab consists of a rotating anode x-ray generator with right and left ports. Each port is equipped with a set of OSMIC focusing mirrors, an R-AXIS IV++ detector, an X-STREAM cryogenic device, and a control computer. A Haskris heat-exchanger cycles water through the x-ray generator tower for cooling purposes. All of the equipment was purchased from Rigaku-MSD of The Woodlands Texas (<http://www.rigaku.com/>).

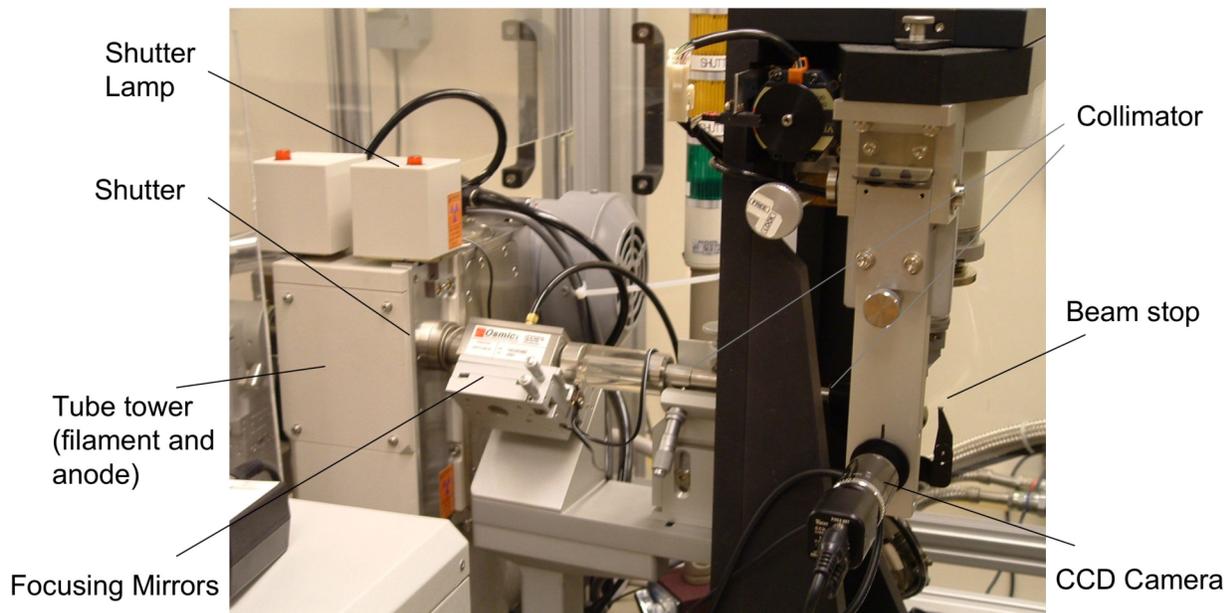


Rotating anode x-ray generator: the large base on which the detectors sit is the Rigaku RU-H3R rotating anode generator. Briefly, x-rays are generated by accelerating electrons from a tungsten filament (cathode) against a copper plate (anode) by a high voltage. As electrons from the filament strike the copper plate, electrons from inner shell orbitals of the copper atoms are ejected, and as outer shell electrons fall into the vacant orbitals, energy is given off in the form of x-rays ($\lambda = 1.54 \text{ \AA}$ for Cu $K\alpha$). The collision generates a lot of heat, which is dissipated by having the anode rotate, and by cycling water through for heat exchange (see description of Haskris below). All of this is going on in the “tube tower” which extends upward from the central part of the generator tabletop. As the x-rays emit from the copper plate, they are captured by a crystal monochromator, allowed to escape through a beryllium window and a shutter that is opened and closed, and focused by mirrors into a $\sim 0.3 \text{ mm}$ i.d. beam through a collimator that is directed at the crystal.

The generator front panel underneath the tabletop has two sides. The right side controls the vacuum. The vacuum inside the tube tower is necessary for optimal generation of x-rays. There is a standard oil-based roughing pump (RP) that creates the initial vacuum, and a turbo molecular pump (TMP) that kicks in to complete the vacuum. The TMP is what makes the jet engine sound when the system is starting up. The ion gauge (IG) reads out the vacuum level; the lower the number in the red lights on the far right side of the panel, the lower the conductivity and the higher the vacuum. A number of around 0.07 is typical during standard operation with the x-rays up to full power.

The left front panel controls the x-rays. When the vacuum gets down to an acceptable level (0.2 or so on the IG), the x-rays can be turned on. Pressing the Target On starts the anode rotation, which is what makes most of the noise in the x-ray lab. Once the target is rotating, the x-rays can be turned on. After turning the x-rays ON, they will initialize at 20 kV 10 mA (voltage/current). There are several warning lights for explanations of when the x-rays automatically shut off for any reason, such as problems with the filament, cooling, target rotation, power, etc. Please see Appendix B for detailed procedures of turning ON/OFF the x-rays.





Osmic focusing mirrors: connected to the tube tower are the Osmic mirrors, which focus the x-rays into a ~ 0.3 mm i.d. beam. The volume inside the mirrors is continuously purged with helium gas (ultra-high purity grade). This is necessary because the He gas is essentially invisible to the x-rays, whereas interaction of the x-rays with O₂ results in oxidation of the surface coating of the mirrors, which is expensive to replace. When collecting data, always check to make sure that the helium tank on your port is not empty. Note: do not confuse the He tank with the He compressor, which is used for the Cold Stream, the two are distinct pieces of equipment for distinct purposes.

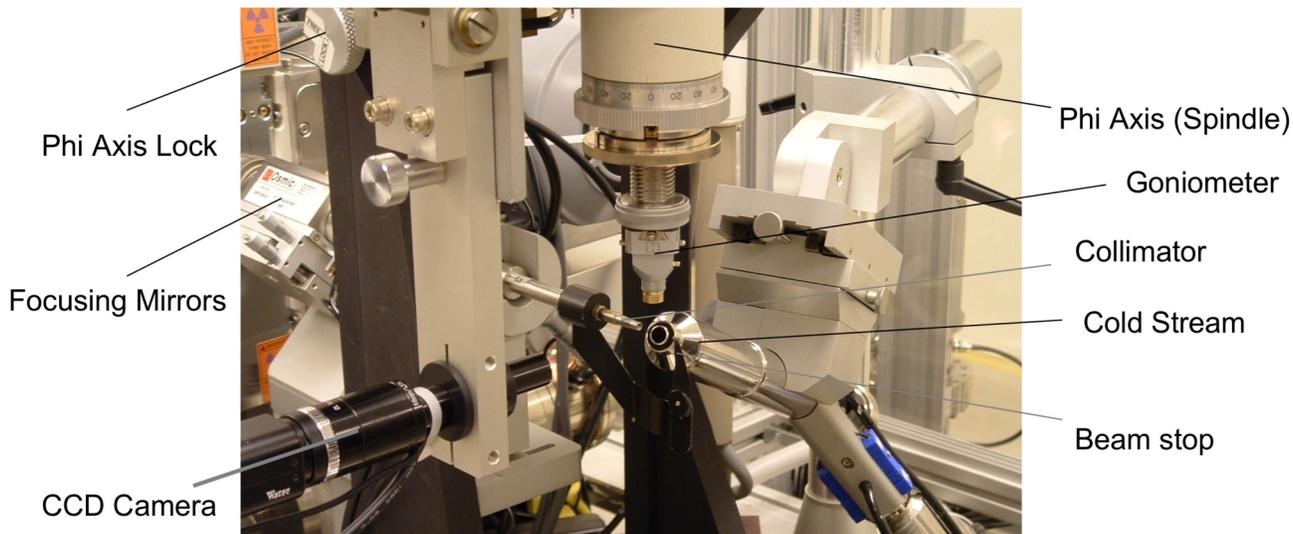
Shutter: You can't see the shutter; it lies at the interface of the tube tower and the mirrors. Basically it is a metal disk that rotates to an open or closed position. When the shutter for one of the ports (right or left) is open, the x-rays are coming out, and as a warning two red lights come on: one on the little cube above the tube tower and one on the "stoplight" that lies behind and to the right of the tube tower. The shutters are controlled manually by switches on the front x-ray panel, which have Open, Close, and Ext. positions. When in the external position (flipped down), the shutter can be opened and closed by the control computer. The shutters can only be opened when all of the doors to the enclosure system are properly closed, unless the shutter interlock is in the override position. Note that the left and right ports have separate enclosure systems so that the left shutter can be open while a crystal is being mounted on the right side, and *vice versa*.

Inverse Phi axis: the crystal is mounted hanging upside down on an inverse Phi axis, which is located immediately after the collimator tube. The crystal rotates about a single axis called Phi. This is mechanically controlled by the computer when in the "lock" position, or can be manually rotated by hand when the rotation is "free", as is needed during crystal alignment.

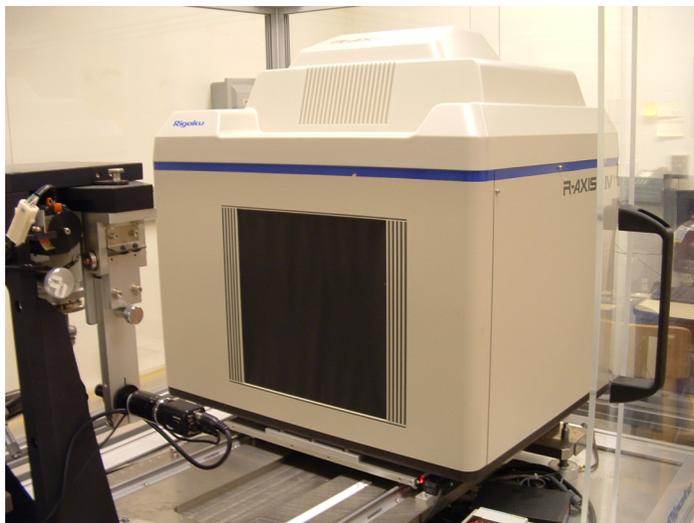
CCD camera: perpendicular to the x-ray collimator tube is a CCD camera directed at the crystal and output to the video monitor. This is used for crystal alignment.

Beam stop: the beam stop is located behind the crystal (from the point of view of the x-ray beam collimator). It stops the direct beam, so that it doesn't reach the detector. The beam stop and the arm

that holds it in place will leave a shadow on the detector that will be observed on each image. If the beam stop were not in place, the direct beam would be observed as a hugely intense spot in the center of the detector, and it would be difficult to erase, possibly damaging the detector. Thus, it is important to always make sure that the beam stop is in place before you begin collecting images. Occasionally it can be knocked out of position slightly. If you ever notice a strong spot near the center of the image (at the edge of the beam stop shadow) stop collecting data immediately and notify the instructor.



Detector: the R-Axis IV++ detector is by far the most expensive piece of equipment in the room. Be very careful not to let anything knock into the front surface of the detector. The detector consists of a phosphorous imaging plate, not very different from the Storm systems you may have used for exposing ^{32}P labeled gels. The image plate is coated with a phosphorous compound that is excited to a long-lived high-energy state by the x-rays hitting the plate at a particular position during image exposure (while the x-rays are being diffracted by the crystal). During image readout, a laser scans the plate in lines (from upper left to lower right), and positions on the plate that were excited by the x-rays during the exposure will give off a signal that is observed electronically. The image plate is divided into an array of 3000 x 3000 pixels, each of which has an associated intensity that is measured. There are actually two image plates within each detector, so one can be exposed while a second is being read out. The “plates” are really flexible belts that rotate around to the expose, readout, and erase stations within the detector. At the erase station, a set of bright lights is quickly flashed on the image plate, to re-set the phosphorous compound to the ground state. The detector sits on a stage, which can be moved back and forth to change the crystal-to-detector distance, which is typically 10-30 cm. The detector on the right port sits on a 2θ stage that can be swung out to the right (toward the front of the generator). This increases the angle that can be observed at a given crystal-to-detector distance.



Control computer: each detector is controlled by a PC running Windows 2000. The images are first captured on the blue box underneath the detector, referred to as the “R-Axis controller”. The data collection process is controlled by the CrystalClear software, which only runs on Windows 2000. The images take up a lot of space, and therefore should be backed up by the 4.8 GB DVD drive and then deleted. The right port control computer has a DVD+RW drive, which accepts a variety of DVD+RW disks, while the left port has a DVD+/-RW drive, which is somewhat picky with regard to which disks it will accept (it likes Sony DVD+RW). There is a separate Linux PC (on the right port side), which is available for data processing using HKL software, but the images first need to be transferred to it via ftp, which is tedious.

X-STREAM: each port has a separate X-Stream cryogenic device, which consists of a nitrogen generator, a cold head, and a helium compressor. The nitrogen generator separates out N₂ from the atmosphere, and sends it to the cold head (located behind the generator) via the thin black plastic tubing that runs along the floor. The helium compressors cycle liquid helium to and from the cold head, which cools the N₂ gas in the cold head to temperatures approaching -180 °C. The nitrogen gas that is cooled is then passed through a nozzle that directs it immediately onto the crystal. There is a separate “Warm Flow” stream of N₂ that does not enter the cold head but instead passes along the outer portion of the nozzle. The warm flow prevents formation of ice at the edge of the cold stream, where it meets the crystal-mounting pin. The temperature settings are entered into a control box that sits on top of the helium compressor. The temperature is also controlled by the “Cold Flow” adjustment on the front panel of the nitrogen generator. The cold flow usually varies from 62-90 psi. A lower cold flow will result in a colder N₂ stream, because the N₂ will spend a longer time getting cooled by the liquid helium in the cold head. If the cold flow gets too low, the N₂ can condense inside the cold head, which snowballs and eventually leads to blockage of the N₂ flow. When this happens, the helium compressor must be turned off for several hours to allow the blockage to warm up and dissipate (the N₂ generator should remain on to flush the system).



Haskris heat exchanger: this is the big grey piece in the back left corner of the room. It has a closed loop of distilled water that cycles through the anode and TMP of the generator, as well as the helium compressors. Inside the Haskris, this closed loop has a storage tank that is cooled by an external water flow for heat exchange. Much as a river is used to cool a power plant, the external water flow absorbs heat from the internal loop. The external flow is controlled by a valve that opens and closes as necessary to maintain the appropriate temperature. The copper



pipes and filter assembly along the wall supply the external flow to the Haskris, and the output feeds into the glass piping that extends along the back wall of the room.

Safety considerations: the direct x-ray beam can cause severe damage to people very quickly. Scattering of direct x-ray beam off of the beamstop can also be harmful. The enclosure system is designed to allow the shutters to be open only when all of the leaded plexiglass doors are closed (there are sensors embedded in the doors). The doors do not allow any detectable x-rays to escape, so the equipment is entirely safe to operate when the interlock system is in place (as is normally the case). However, it is possible to override the interlock system with a special key, which is necessary for doing routine beam alignment, etc (note that this should only be done by authorized personnel). When the interlock system is in the override position, the shutter will remain open even if the doors are open, in which case a user could stick their hand in the direct beam and become exposed. Therefore, all users should test to make sure that the interlock system is operating correctly. This is done by opening the shutter when all of the doors are closed, and then opening a door and checking to see that the shutter closes automatically. After this test, the user will know that the interlock system is in place, and it should not be possible for the shutter to be open while the user is mounting or aligning a crystal. For general practice, however, it is always a good idea to keep ones hands out of the path of the direct beam at all times.

APPENDIX A

Checklist of Do's and Don'ts

1. Always make sure that the enclosure interlock system is functioning properly by checking to see that the shutter closes when a door is opened. This should be done at the beginning of each data collection session.
2. Always move the detector back all the way (~450 mm) before mounting or aligning a crystal. Getting it out of the way minimizes the chances of knocking something into it.
3. Always make sure the beam stop is in place before collecting data. Taking images without the beam stop in place (and correctly aligned) could potentially overexpose the detector. Occasionally the beam stop might get out of alignment, and you might notice an intense spot at the edge of the beam stop shadow. When this happens stop collecting data and notify the appropriate person to fix the beamstop.
4. Always make sure that the helium tank for the port (right or left) you are using is not empty before opening the shutter to begin collecting data. Operation of the x-ray beam when there is no helium purging through will result in oxidation of the coating of the mirrors, which is very expensive to fix.
5. Don't change the settings on the Control Box for the X-stream unless you know what you are doing.
6. Back up and delete your image files after you collect data. If the hard drive gets too full, then people can't collect data, in which case they may have to delete other people's data to create space on the drive. It is each users responsibility to backup and delete their own data promptly.

APPENDIX B

Procedures for operation of RU-H3R X-ray generator

1. Starting up generator from OFF status.

Press "System Start" button on front right vacuum panel of generator. You will see the RP (roughing pump) light come on, followed by the TMP (turbo molecular pump), IG (ion gauge), and finally the operate light, in that order. The ion gauge creates the initial vacuum, and the TMP finalizes the vacuum. The ion gauge comes on to measure the vacuum level based on conductivity. A lower number for the IG (red numbers) means a better vacuum. The operate light comes on when the IG reads about 0.25.

Once the operate light is on and the vacuum is down to about 0.1, press the X-ray panel "Power ON". Next press the "Target ON" button, which will begin the anode rotation, and then you should see the "Ready" light come on (both on the front panel and on the stoplight). You might see a slight jump in the IG reading, since as the anode begins to rotate, it might stir up some air pockets that momentarily increase the vacuum. Once the vacuum has stabilized, press the "Xray ON" button, which will turn on the voltage across the filament and the anode, and x-rays will begin being produced inside the tube tower.

The power readings will begin at 20 kV 10 mA. Normal operation is at 46 kV 90 mA. It can be operated as high as 50/100, but we tend to get shut offs (with OL alarm) at these settings. The power is raised by first slowly increasing the kV from 20 to 46 over a period of ~5 minutes. Tap the up key in individual increments, rather than holding down on it. Next, slowly increase the mA from 10 to 90, in a similar manner. Once the power is up to 46/90, you are ready to begin.

2. Recovering from a generator alarm.

Sometimes the x-rays shut off automatically when there is a problem. You will hear a loud and annoying alarm, and you will likely see one of the 10 alarm lights illuminated. This will happen if the filament is blown out (just like a lightbulb), or for various other reasons. Before hitting the “Reset” button to turn off the alarm, notice which alarm light is illuminated, as this will give you information as to what the problem is. Here are the definitions of the alarm lights:

FC	Filament current. The filament may be blown out.
TR	Target rotation, there is a problem with the rotation of the anode.
CW	Cold water, there is a problem with the temperature getting to high or cold water flow.
OL	Overload, there is a problem with the power supplied to the unit

After hitting the “Reset” key, you can try to start up the x-rays again. If it is an OL alarm, then usually you can restart. If it is FC, TR, or CW, then there is probably a problem that will require maintenance. First notice if everything is OK on the Vacuum panel (check to see that the operate light is on). If not then hit “Reset” on the vacuum panel, and then “System Start”, following procedure (1) above. Make sure that vacuum starts up normally and that the operate light comes on. If the vacuum panel is OK, then press the “Target ON” and “Xrays ON” buttons of the x-ray panel as described above. The x-rays will come on again to 20/10, and the power can be increased as described above.

If the system soon shuts off with another alarm, then it is likely that there is a problem (such as blown out filament) that will need to be corrected by the instructor.

APPENDIX C

Procedures for operating X-STREAM cryogenic device

I. General Operation:

It is OK to leave the Cold streams on when they are not in use. If you think that nobody will be using the cold stream for several days, it is a good idea to turn off the He compressor, and then the next day it is OK to turn OFF the N2 generator. Note: the N2 generator should always be ON when the He compressor is ON or has recently been on such that it is still cold inside the cold head. This means that you should never turn OFF the He compressor and the N2 generator at the same time, always leave the N2 generator running until the temperature inside the cold head has warmed up to ~ 22 °C. Otherwise, ice will form in the cold head. The ice will melt and leave H2O that can sit and possibly damage the seals inside the cold head. Similarly, never turn the He compressor ON unless the N2 generator has been ON for at least 2 hours.

I. Turning X-STREAM ON:

1. Turn on N2 generator, let it run for ~2 hours.
2. Turn on He Compressor, temp will go down to -180°C.

Note: you should not need to change any of the settings on the Control Box. In fact, you should not adjust these unless you really know what you are doing. You may need to hit left arrow key on the Control Box to toggle through to get to the display that will read out the temperature.

II. Turning X-STREAM OFF:

1. Turn off He Compressor, wait ~24 hours for temperature to warm up to ~25 °C.
2. Turn off N2 Generator.

Note: it is important to leave the N2 generator on so that N2 gas will be pushed through while the Cold head warms up. Otherwise, ice will form in the cold head, and after the ice melts water will sit inside the cold head, possibly leading to corrosion.

III. What to do when cold head gets clogged:

After the X-STREAM has been on for several days, it may get too cold such that N2 condenses inside the cold head, and eventually it will get clogged. You will notice that the temperature on the Control Box reads -180°C (it never gets lower than that), and there is a number on the bottom right that is rapidly fluctuating from 0-25 or so. This is the heater being turned on to try to counteract the freezing. The problem is evident when the N2 cold flow (on the front panel of the N2 generator) gets low, due to the fact that it is clogged. So if you see the N2 flow get low (lower than 60 psi), it is clogged and eventually there will not be N2 flowing onto your crystal. Note that the temperature on the control box is read inside the cold head, not at the crystal.

To unclog the X-Stream:

1. Turn off the He compressor, leave the N2 generator on.
2. Wait ~24 hours for the cold flow to go back up to normal (65-80 psi).
3. Turn He compressor back on.

Note: the He compressor should never be on when the N2 generator is not on. The N2 generator should always be on when the He compressor is on or was on recently such that the temperature has not stabilized.

IV. Tuning the X-stream temperature.

If the x-stream is getting repeatedly and quickly clogged after turning on the He compressor, then it is necessary to make an adjustment to increase the target temperature. This is done by simply increasing

the cold flow on the N₂ generator. Do not change the settings on the Control Box that sits on top of the helium compressor. Note that a higher cold flow actually raises the temperature because the N₂ gas goes through the cold head more quickly (i.e. spends less time getting cooled). If it is set to for example 70 psi, then try turning it up to 72 psi, wait a couple of hours, and see where the temperature re-stabilizes at. A good target temperature is a few degrees above -180°C, i.e. -178°C. If this does not work (i.e. if the temperature remains too cold), there may be a problem with the purity of the N₂ coming from the N₂ generator. As the O₂ level gets higher (decrease purity of N₂), the gas is easier to cool and freezing out becomes more of a problem.