# System Cool-down and Operating Procedures<sup>1</sup>

# I. What to do at room temperature.

#### 1. Pump the OVC.

Pump the OVC through the OVC pumping port to  $2 \times 10e-7$  mBar using the turbo pump. If the OVC is not vented before, pump for about 6 hours should be enough. If the OVC is vented, pump for 24 hours/overnight.

#### 2. Pump the IVC.

Pump the IVC (there are two valves, pump both) through the IVC pumping port to  $2 \times 10e-7$  mBar using the turbo pump.

#### 3. **Pump the sample space**.

The sample space should be under vacuum from last run but should be checked. Use the turbo pump to pump the sample space to  $2 \times 10e-6$  mBar. If the probe is not loaded, pump through the top of the sample space gate valve. If the probe is already loaded, pump through the sample space pumping port and the probe sliding seal port (pump the vacuum lock above the gate valve first before opening the gate valve). Pumping the sample space typically takes about 30 minutes.

# 4. Check the vacuum in OVC and IVC.

Connect the OVC pumping port to the turbo pump, run the pump to below  $2 \times 10e-7$  mBar, open the valve and see if the pressure of OVC goes up compared to the vacuum obtained by the first pumping. If it does, keep pumping and check again, making sure the rise of pressure comes from gas-out from the inside materials of the OVC, not from a leak.

Do the same check for IVC.

During later stages, the vacuum of OVC and IVC can always be checked/pumped at proper times.

#### 5. Pump and Flush 4He bath with N2 gas.

Before pump the 4He bath, make sure the OVC is under vacuum (see previous section of 4). Otherwise the 4He bath may collapse.

(1) Pump 4He bath. (10 min.)

Close the 4He recovery line valve on the top of the recovery meter and use the big mechanical pump to pump the 4He bath through the 4He bath exhaust port in order to remove air and moisture. Be careful and open the small valve on the pump first. When the vacuum gauge on the pump goes up to 25 it's OK to open the gate valve on the pump. The vacuum can be monitored by the mechanical gauge connected on the 4He exhaust line or by the Motorola 4He electric pressure sensor. A rough vacuum of about 20 mBar should be OK.

<sup>&</sup>lt;sup>1</sup>Refer to "sensors.doc" for sensor information and temperature measurement of the system.

(2) Flush 4He bath with N2 gas. (10 min.)

Stop pumping and close the valve of 4He bath exhaust line. Connect one end of a rubber hose to liquid Nitrogen tank gas use port and open the gas use valve. Feel the gas flow rate on the other end and adjust the valve if it's too slow or too fast. Connect the other end of the rubber hose to the 4He bath port with a small valve, open the valve and flush the bath with N2 gas to atmospheric pressure. Watch the mechanical gauge on 4He exhaust line or the Magnehelic gauge, but keep in mind the Magnehelic gauge gives differential pressure relative to atmospheric pressure. It should be enough if the gauge reads about 10 inch of water. 1 inch of water is 2.5 mBar.) Close the small valve on 4He port before close the gas use valve on the liquid nitrogen tank.

# 6. Pump 1K pot and check 1K needle valve.

Before pumping the 1K pot check that the needle valve is closed. Proceed to pump the sample space for 10 minutes before continueing. Use the big mechanical pump to pump the 1K pot through the 1K pumping port. A rough vacuum of below 10 mBar should be OK. Be careful and open the small valve on the pump first. When the vacuum gauge on the pump goes up to 25 it's OK to open the gate valve on the pump. Stop pumping and keep the 1K needle valve closed while the system is being cooled. In order to check if the needle valve of 1K pot works, slowly open the needle valve after the 1K pot is pumped and send N2 gas from the 4He bath. Watch how the pressure of 1K changes from the Motorola 1K electric pressure sensor. Pump 1K pot again after the needle valve is checked.

# 7. Pump LPR<sup>2</sup> and check LPR needle valve.

Pump the lambda point refrigerator and close the valve in the pumping line. Open the needle valve on the lambda point refrigerator and watch the pressure gauge. Check that the pressure rises quickly to approximately atmospheric pressure. Pump the LPR again after the needle valve is checked. Keep the LPR pumping port and the needle valve closed as the system is being cooled.

# II. Cool the system to 77K.

# 1. Fill the 4He bath with liquid nitrogen. (3 hrs.)

Replace one of the two 4He bath relief valves with a Tee valve for vent when filling the bath with liquid nitrogen.<sup>\*</sup> Close the bar valve. Block the upper end of the liquid nitrogen blow out tube with a rubber stopper and fit the tube into the 4He bath siphon entry and screw the tube into the siphon cone in the bath. Do not over tighten the tube. Connect liquid nitrogen transfer rubber hose to the liquid port of a liquid nitrogen storage tank. Remove the rubber stopper on the blow out tube and connect the other end of the transfer rubber hose to the blow out tube using a clamp. Check the pressure of the storage tank and depressurize it to below 10 psi before transfer. Open the liquid nitrogen tank liquid port valve to start filling liquid nitrogen into the bath. For the filling rate,

<sup>\*</sup>Optional: Can connect hose from Tee valve to nitrogen jacket filling/vent ports to pre cool the jacket before LN<sub>2</sub> transfer to jacket.

<sup>&</sup>lt;sup>2</sup> LPR stands for lambda point refrigerator.

temperature decreasing rate of 1K/min should be reasonable. To monitor the temperature of 4He bath, one can measure resistance of the AB sensor<sup>3</sup> mounted on top of the magnet using the Keithley multi-meter. Refer to Table 1 on page 5 for sensor information and refer to system manual page 27 for Resistance-Temperature relation of AB sensors. After exchange gas is introduced into IVC other temperature sensors can be used to monitor temperature of insert/sample space as well (see next section). It may take about one and half an hour for the liquid nitrogen to go above the top of the magnet. There's another AB sensor 10 cm above the top of magnet. After these AB sensors read about 77 K and before liquid goes up to the siphon cone, one can stop filling, unscrew the tube out of the siphon cone and pull the tube up for a few inches, and then start filling again. In general another one hour or so should put enough liquid nitrogen into 4He bath for pre-cooling the system. (It's not necessary to fully fill the bath.) After filling the bath and add exchange gas into IVC (see next section), another hour should be enough for the system to be pre-cooled.

# 2. Add exchange gas of 3He (or 4He) into IVC and cool the sample space to 77 K.

Admit about 10cc of 4He gas from the 3He cylinder into IVC through the IVC pumping port with a small valve. (The advantage of using 3He over 4He is it's easier to be pumped out later.) Wait for the system to cool down. The sorb temperature can be monitored by  $ITC^4$  from the AB sensor connected to it. If the probe is already loaded to it's pre-cool position (see section II-4), the platinum sensor mounted on top of sample can also be used to monitor temperature. Refer to the file "sensors.doc" for temperature sensor information and ways of measurement. (The low temperature sensors of RuO2 connected to 1K and 3He tail can not be monitored by ITC if the temperature is above 7 K. If needed, one can use Keithley 2000 2R measurement to measure the resistance of these sensors. The room temperature resistance is about 2.21 K $\Omega$ . When the resistance increases to above 2.46 K $\Omega$ , the system should be at 77 K.) Typical enough pre-cooled temperatures might be: sample platinum sensor 90 K, sorb AB sensor 100 K. Temperature may not get all the way down to 77 K, 150 K may be sufficient.

# 3. Fill the liquid nitrogen jacket with liquid nitrogen.

After the 4He bath is filled with liquid nitrogen the nitrogen jacket can be filled. Use rubber hose to connect one of the nitrogen jacket filling/venting ports with liquid nitrogen tank liquid port. Keep the other filling/venting port open and put a piece of rubber hose extension on it for venting. Fill the nitrogen jacket to a level of about 20%. The liquid nitrogen in 4He bath will be used to continue filling the nitrogen jacket after the system is pre-cooled. It may take 2 hours for the liquid nitrogen level on ILM to start reading the level.

# 4. Load the sample probe for pre-cooling.<sup>5</sup>

The probe should be loaded at this point if it hasn't been done. Be sure that the contacts are all grounded before cooling, should also be the case for changing of samples.

<sup>&</sup>lt;sup>3</sup> AB stands for Alley Bradley temperature sensor.

<sup>&</sup>lt;sup>4</sup> ITC is the temperature monitor/control unit of the system.

<sup>&</sup>lt;sup>5</sup> If the sample is mounted and necessary contact test at R.T. has been done, all contacts should be grounded using LabView program while the system is being cooled.

Keep the gate valve closed and load the probe above the gate valve. Use the turbo pump to pump the volume above the gate valve through the sample space pumping port and the sliding seal pumping port. Pump to  $2 \times 10e-6$  mBar. Besides reading from the turbo pump controller, the vacuum can also be monitored using the 3He mechanical gauge or the 3He Motorola electric pressure sensor to monitor the pressure. When the desired vacuum is obtained, close the sample space pumping port valve while keep pumping through the sliding seal pumping port. Push the sample probe down until the copper cone is approximately 50 cm above the contact on the 1 K pot, and use the clamp to hold it in this position while cooling down the system. (Only when the insert is cold is any <sup>3</sup>He exchange gas allowed into the sample space. Until that time, the probe cools by radiation alone.) This position is chosen for the following reasons.

- a) If the probe is not inserted, the thermal radiation down the central access will prevent the <sup>3</sup>He pot and 1 K pot from cooling properly.
- b) If the probe is pushed all the way into the system, the probe itself will hold the lower parts of the insert at a high temperature because it does not cool as quickly as the insert.

The temperature close to sample (or probe end) can be monitored by the speer sensor or platinum sensor mounted close to sample holder.

#### 5. Add exchange gas of 3He into sample space and pre-cool the sample space.

Admit 3He gas from the dump into the sample space through the 3He port, if "3He" pressure is  $\sim$ 25 mBar. The correct order to do this is: open the dump valve, close the dump valve, open the 3He port valve, and close the 3He port valve.

Monitor the temperature of the speer sensor or the platinum sensor and cool the sample space down to about 100 K.

# 6. Blow out liquid nitrogen from 4He bath to nitrogen jacket.

After the system is pre-cooled, one needs to transfer liquid nitrogen from the 4He bath to the liquid nitrogen jacket. Close the 4He bath exhaust port valve and block the check valve using a rubber stopper if the check valve is fitted. Screw the liquid nitrogen blow out tube into the siphon cone. Use rubber hose to connect the top of the blow out tube and one of the nitrogen jacket filling ports (keep the other filling port open with a rubber hose extension fitted for exhaust). Use another rubber hose to connect the 4He bath port (with a small valve fitted) with gas use port on liquid nitrogen storage tank to blow liquid nitrogen from 4He bath to the nitrogen jacket. The right order of doing so is: Connect rubber hose to gas use port on storage tank, open the valve and feel the gas flow on the other end of rubber hose, adjust valve if flow if too high or too low, connect the rubber hose to nitrogen jacket, and open the small valve. A pressure of 200 mBar above atmospheric pressure in the 4He bath should be sufficient to blow nitrogen out.

The following signs may tell there's no liquid nitrogen blown out any more:

- The liquid nitrogen level on ILM doesn't go up any more;
- The pressure of 4He bath drops to atmospheric pressure;
- The metal part of the blow out tube nearest to the cryostat is no longer wet on the outside;

After the 4He bath is empty, unscrew and take the blow out tube out and seal the 4He siphon entry.

Bring the liquid nitrogen level up to about 80%. If the liquid nitrogen from 4He bath is not enough, fill the nitrogen jacket with liquid nitrogen from a tank.

# III. Cool the system to 4.2 K: Preparations and transferring liquid 4He.

1. Fit a non-return valve to at least one of the liquid nitrogen jacket ports, and either a factory supplied relief valve or a Bunsen valve to the other ports. This will ensure that air is not condensed into the tubes during the liquid helium transfer, causing dangerous blockages.

# 2. Pump and flush the 4He bath with N2 gas.

Check that the 4He recovery line valve on the top of the recovery meter. Use the big mechanical pump to pump the 4He bath through the 4He bath exhaust port. The pressure should fall steadily to about 20 mbar (Motorala 4He) or 30 mBar (mechanical gauge). If this does not happen (for example, the pressure hesitates at 100 mbar) it may indicate that the liquid nitrogen has not all been removed. Vent the bath with N2 gas and try to blow out any remaining liquid.

After making sure there's no more liquid nitrogen in the bath, stop pumping and close the valve of 4He bath exhaust port. Connect one end of a rubber hose to liquid Nitrogen tank gas use port and partly open the gas use valve. Connect the other end of the rubber hose to 4He recovery port and flush the bath with N2 gas to atmospheric pressure.

# 3. Pump and flush the 4He bath with 4He gas.

**P**ump the 4He bath again to about 20 mBar (Motorala) and flush the bath with 4He gas to atmospheric pressure, for about 10 minutes.

# 4. Check Sorb and 1K temperature (should be close to 77 K).

Temperature close to 77 K should be obtained in order to make the transfer and use of liquid 4He efficient.

# 5. Transfer liquid He into 4He bath.

Before starting transferring, make sure the recovery line is connected to the system and the recovery line valve above the small mechanical pump in the pump room is open, should also be sure that the 1K is connected to the recovery as well. Leave Tee port open for vent when transferring. For initial transfer put tube in directly without waiting for liquid to come out. Optional: Pump liquid He transfer tube. Refer to manual for general guidelines of transfer and refer to file "Helium Transfer Procedures.doc" for detailed instructions on warm and cold transfer. Some important points of transfer are listed here:

- The transfer tube needs to be cooled enough before the tube is put into the cryostat, case only for refilling not for initial transfer.
- For initial warm transfer the tube should be put all the way to the siphon cone. As liquid goes up to top of magnet (Use AB sensors to monitor temperature so get an idea of where the liquid surface is.), the tube should be pull out a few inches so its end is not in the liquid. When pressure starts to increase and transfer starts to slow pull transfer tube out.
- The pressure in the cryostat and storage dewar should be checked frequently. The pressure in the storage dewar needs to be 2 to 3 psi for efficient transfer.
- The value of T<sub>1</sub> shouldn't be much lower than 266.0 K. This problem would signal to fast of a transfer.

During the early stage of warm transfer before liquid level goes to the lower end of liquid 4He level probe, the level of liquid can be monitored roughly by measuring the resistance of the Allen Bradley sensors mounted around the magnet and the LPR. The location and resistance of the sensors are as follows (Table 1)<sup>6</sup>

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	Location	Connector color	Resistance $(\Omega)$			
			R. T.	77 K	4.2 K	4.2 K Measured
R1	$10 \text{ cm above } \text{LPR}^7$	Red-Yellow	158	179	961	952
R2	On LPR	Red-White	156	177	927	926
R3	Top of magnet	Red-Blue	160	183	937	940

# Table 1 Allen Bradley sensors in 4He bath.

During the transfer, temperatures of the sorb (Allen Bradley sensor, ITC sensor 1 input), 1K (RuO2 sensor, ITC sensor 3 input), sample (platinum sensor measured with Keithley 2000, see sensor.doc, and RuO2 sensor, measured with ITC sensor 2 input) should all be monitored.

# IV. Condense 3He, cool to base temperature and run the system at base temperature.

# 1. Add 3He gas into the sample space.

When temperatures of the sorb and 3He tail (or sample) drops to 10 K or so, open the <sup>3</sup>He port valve on the insert and open the valve on the cryopump to put 3He gas into the sample space. Should be ~500 mBar on dump before putting 3He into sample space. Decreases by 170 mBar each time when adding then wait until pressure is 100 mBar. The pressure of the sample space should increase to about 100 mBar. Close the 3He port valve on the insert. (Make sure the valve on the <sup>3</sup>He dump remains open at all times during operation of the <sup>3</sup>He refrigerator. The <sup>3</sup>He valve on the insert has a pressure

<sup>&</sup>lt;sup>6</sup> During transfer, the resistance may saturate at about 700 Ohm when the temperature is close to 4 K.

<sup>&</sup>lt;sup>7</sup> LPR stands for Lambda Point Refrigerator.

**relief function and will safely vent** <sup>3</sup>**He gas into the dump if there is any accidental heating of the system.)** During this time the sorb temperature may rise to about 30 K. Wait for the 3He gas to enter the sorb. After the pressure of the sample space drops to 20-30 mBar, open the <sup>3</sup>He valve on the insert to allow <sup>3</sup>He gas to enter the sample space, and close the valve. When the pressure of the sample space goes down to 20-30 mBar, open the 3He port valve on the insert again and add more 3He gas from the 3He dump. This need to be repeated a few more times in order to bring the pressure of the dump below 20-50 mBar, dump will read 0 mBar, and so most of the 3He gas goes into the sample space (or the sorb before condensing).

#### 2. Pump out exchange gas in IVC and assist the cool down.

After the sorb/sample temperature goes to 20 K, one can start pumping out the exchange gas in IVC using the turbo pump. Pump the IVC to  $2 \times 10e-7$  mBar (may need to pump overnight if 4He was used as exchange gas).

The needle valve of 1K can now be partly opened **WITHOUT PUMPING** the 1K pot in order to assist the further cool-down of the system. (Do not cool the 1K below 4.2 K while pumping the exchange gas in the IVC out as the cold surface of 1K will retain some of the gas, and this may create an unacceptable heat load later.) If the system already cools close to 4 K before IVC is pumped, close need valve in order to slow down the cooling.

#### 3. Condense 3He.

Close the IVC pumping port valve and stop pumping when the desired vacuum is obtained ( $\sim 2 \times 10e-7$  mBar). Start pumping the 1K using the big mechanical pump. The 1K pressure should be around 5 mBar (Motorala) 10 mBar (mechanical gauge) all the time when it's being pumped. From now on the 1K needle valve needs to be adjusted in order to get 1K temperature as close to 1K (typical value is 1.5 K) as possible so to maximize the amount of liquid 3He condensed.

The probe should currently be at its pre-cool height (see previous section of II-4). If it's not, it should now be loaded to this position following the procedure described in section II-4. Check and make sure that the sample temperature is below 10K. Remove the holding clamp and lower the probe slowly until the copper cone on the probe meets the 1K pot.

Set the sorb temperature to 32 K, turn ITC control on in the program and let the sorb release 3He gas and start condensing 3He. (Do not use the "auto" option of sorb heater at the beginning of condensing; otherwise the heater will heat the 1 K too much.) Note that the temperature of the 1 K pot will be heated up to about 2 K and then drops. The pressure of the sample space should increase to about 250 mBar and then keeps dropping as 3He is condensed. When the sorb temperature goes up to 20 K set the sorb heater to "auto". 1K will be heated by the heater again so its temperature goes up and down again. When the sample space pressure goes down to 150 mBar, change the sorb temperature set to 27 K and turn 1K needle valve off (turn off cooling on the sorb). The final vapor pressure of condensed 3He depends on the condensing temperature (about 80 mBar at 1.5 K). Refer to the relation between vapor pressure and 3He liquid temperature (Igor file

"Helium Vapor Pressure.pxp"). When the sample temperature goes flat and the <sup>3</sup>He pressure reaches its vapor pressure, wait for about 10 minutes. This should be the end of the condensation, sorb T~25 K for slow pumping results in 3He T~1.5 K.

# 4. Cool sample space to base temperature (~ 1 hour).

When all the <sup>3</sup>He has condensed the sample space can be cooled to the base temperature (~ 0.2 K). Turn off the sorb heater and let the sorb cool down. The cooling down can be assisted by opening the 1K needle valve to 1/16 turn (turn on cooling on the sorb). The sorb will start pumping <sup>3</sup>He gas when its temperature drops to about 15 K. If there is sufficient flow of 4He through the sorb heat exchanger, the final sorb temperature will drops to about 2.5 K. At this point the <sup>4</sup>He flow needs to be reduced to 0.5 - 0.8 liters/min. This flow should be sufficient for operation at base temperature. Monitor the 1K, 3He port and sample temperatures. The <sup>3</sup>He pot and sample should cool rapidly to about 0.4 K and then cool to base temperature in about an hour. Without extra heat loading such as from microwave, the system should be able to hold at the base temperature of about 0.25 K for 90 hours.

# 5. Re-condense the 3He.

When all the <sup>3</sup>He has evaporated the sample will begin to warm up. The liquid needs to be re-condensed in order to get base temperature again. The procedure of re-condensing is the same as described above except that the probe should remain in its fully loaded position.

# 6. Remove probe when the system is cold (change sample).

At the end of each experiment run, the <sup>3</sup>He must be pumped into the sorb or removed from the cryostat into the dump using the cryopump (see section V-2), so that only a negligible amount of 3He gas is left in the vacuum lock when taking the probe out. The sorb is capable of reducing the pressure to below  $10^{-4}$  mbar if it is below 8 K. When the <sup>3</sup>He pressure in the cryostat is sufficiently low, ~80-100 mBar, the probe may be removed. (The pressure can be monitored using the mechanical gauge on the probe or the 3He Motorola pressure sensor.) Check that the valve on the vacuum lock of the probe and the <sup>3</sup>He valve on the insert are both closed. Make sure that the gate valve is fully open. Pump the probe sliding seal using the small orange pump. This will pump away any air that leaks through the outer seal as the probe is moved. Set the Sorb heater to ~25 K to be sure that there is exchange gas available to help the probe cool while being removed. The sample probe can now be lifted slowly out of the cryostat. It will take about 10 to 15 minutes to withdraw the probe. This is most conveniently done in several stages. A clamp is provided to hold the probe at various positions during this procedure. If the lower part of probe just taken out feels cold, one should stop and wait for it to warm up. If you see any condensation on the tube as it emerges from the seal assembly, then the probe is removed too quickly. If the 'O' rings in the sliding seal are frozen, air may go into the sample space. When the probe has been withdrawn completely<sup>8</sup>, turn off the Sorb heater and wait for the <sup>3</sup>He pressure to drop below 80mBar at which point one can close the gate valve carefully (in case the probe is not fully withdrawn); it should close with a click.

<sup>&</sup>lt;sup>8</sup> The length of the tube out of the vacuum lock up to the top of probe should be 173 cm long.

The sample can now be left to warm naturally to room temperature or use dry nitrogen exchange gas through the small valve on the vacuum lock of the probe. Avoid using <sup>4</sup>He as exchange gas because of the risk of contaminating the charge of <sup>3</sup>He. Disconnect the pumping line from the probe. When the sample is at room temperature remove the probe assembly by disconnecting the Klein flange on top of the gate valve.

# 7. Inserting the sample probe when the insert is cold.

Connect the probe assembly to the gate valve. Pump the vacuum lock through the valve on the probe and then close the valve. Start pumping the sliding seal port to prevent the ingress of air as the probe is lowered down. Open the gate valve, pressure should be 13 mBar but 30 is okay and sorb of 15 K. Use the clamp to hold the probe in this position. Check that the valve V2 to the dump is open and that the <sup>3</sup>He valve on the insert is closed. Warm the sorb slightly (set sorb temperature to 10 K then 15 K then 25 K in steps allowing it to semi-equalize) to introduce a few mbar of <sup>3</sup>He exchange gas into the sample space (monitored on the pressure gauge on the probe or the 3He Motorola pressure sensor). Lower the probe slowly until the copper cone is approximately 30 to 50 cm above the contact on the 1 K pot (better to do it in a few steps), and allow the sample to cool to below 20 K before pushing the probe down into final position. Inserting the probe will warm the 1 K pot<sup>9</sup>. It might be necessary to increase the flow of <sup>4</sup>He through the 1K pot to cool it down again. (Open the 1K needle valve more) Allow the whole <sup>3</sup>He charge to condense fully into the <sup>3</sup>He pot. With some experience, you may prefer to use a slightly different procedure to improve the efficiency of the top loading process as follows. Provided that care is taken, there is no reason why the <sup>3</sup>He should not be condensed into the <sup>3</sup>He pot before the gate valve is opened. This allows the probe to be cooled gradually as it is pushed slowly into the insert. If you use this technique you must ensure that the valve V2 to the dump vessel is open and that there is no risk of pumping <sup>3</sup>He away if the sliding seal or one of the valves leaks. For this reason, the procedure described first will be the most appropriate for inexperienced users.

# V. Warm up the system

# 1. Preparations

Before starting to warm up the system make sure it is safe to do so. Make sure that there are no trapped volumes of liquid, gas or condensed solids inside the system, and that all closed volumes with cryogen/cold gas are free to vent or that they are pumped continuously as the system warms up. Prepare these parts of the system as follows:

- The superconducting magnet must be de-energized/Power off.
- The Lambda point refrigerator must be closed down and pumped out (and pumped continuously during warm-up) or vented to the main helium reservoir.
- The 1K pot must be vented or pumped continuously during warm-up.

# 2. Take 3He gas out of sample space.

<sup>&</sup>lt;sup>9</sup> The platinum sensor mounted near sample may saturate at 110 K for low temperature.

Ensure that the <sup>3</sup>He storage vessel is connected to the cryostat and that the 3He valve on the insert and the dump valve are open. Close the 1K pot needle valve and pump 1K pot. The <sup>3</sup>He should be removed with the cryostat between 4.2 K and 80 K, to ensure that any air or water that has entered the system is removed from the gas. Heat the sorb to about 50 K. Apply heat to the <sup>3</sup>He pot to boil off any remaining liquid <sup>3</sup>He. (50 mW should be sufficient to remove all the liquid in a few minutes.) The pressure in the <sup>3</sup>He storage vessel should begin to rise as the <sup>3</sup>He leaves the cryostat. When an equilibrium pressure is reached, the <sup>3</sup>He remaining in the insert can be removed as follows. Open the valve on the cryopump. Wait until the pressure in the dump equalizes with the pressure in the insert and then close the valve on the dump. Slowly lower the cryopump into liquid helium at 4.2 K and keep the valve on the top of the cryopump open. After about 10 minutes, the remaining <sup>3</sup>He gas in the insert should be at a lower pressure. Close the <sup>3</sup>He valve on the insert and open the valve on the dump. Keep the cryopump valve open and lift the cryopump slowly to the top of dewar. Check that the <sup>3</sup>He gas in the cryopump is expanding into the dump by observing the dump pressure gauge. When the pressure in the dump stabilizes, close valve on dump and slowly lower the cryopump into the dewar again. After 5 minutes open the <sup>3</sup>He valve on the insert to pump another portion of the <sup>3</sup>He charge out of the insert. Close the <sup>3</sup>He valve on the insert, open valve on dump and lift the cryopump up again. This procedure must be repeated until the dump pressure no longer increases as the cryopump is warmed up. This means that all the <sup>3</sup>He gas has been pumped out of the insert. Cool the cryopump to 4.2 K again and collect the <sup>3</sup>He gas from the connecting lines. (Make sure that valve on dump and the <sup>3</sup>He valve on the Insert are closed.) Close the valve on the top of the cryopump while the cryopump is cold and remove the cryopump from the 4He dewar. The  ${}^{3}$ He gas from the connecting line will be stored in the cryopump. (For safety reasons, it is recommended that all the <sup>3</sup>He gas is stored in the dump, except the gas that is removed during final cryopumping of the connecting lines. If too much <sup>3</sup>He gas is in the cryopump when it is warmed up to room temperature, dangerously high pressures may be produced.) At the end of the first run, the <sup>3</sup>He dump pressure will be slightly less than at the start, as a small amount of gas is stored in the cryopump. This gas can be condensed back into the cryostat for the next experiment at the same time as the dump 3He gas is condensed. On subsequent runs the final pressure should not be significantly different from each run.

# 3. Warm up the system to room temperature.

After the 3He gas has been removed from the sample space, the system may then be warmed up to room temperature. Again, ensure that any volumes that contain cold liquid or gases (including exchange gas) will not be pressurized as the temperature rises. Between runs, the sample space should be kept under vacuum to keep the charcoal in the sorb as clean as possible.

# 1) Warm up the system naturally.

When the system has been prepared properly it can be left to warm up naturally. When the cryogens have all evaporated the system will warm slowly to room temperature. Temperature can be monitored using the sorb AB sensor or sample platinum sensor. If you do not need to use the system again soon this is the easiest way to warm the system up.

2) Warm up the system quickly. If one wants to warm up the system more quickly one can blow out the cryogens and break the insulating vacuum in the outer vacuum chamber. The liquid helium and liquid nitrogen can be blown out of the system into storage vessels. The system will then begin to warm up.