3. *Running the system*

Warning. Make sure that you have taken the necessary precautions to ensure your own safety and the safety of other people working near you.

The following procedure assumes that you are using liquid helium with the system. This cryostats can also be used with liquid nitrogen, but some of the techniques are different. Please see section 3.6 for details.

Ensure that the cryostat's insulating vacuum and the transfer tube have recently been pumped to high vacuum. Connect the system together and prepare it as described in section 2.2.

3.1. *Cooldown: version with low temperature entry arm*

Make sure the needle valve on the transfer tube is fully open. (See section 1.2.) Open the needle valve on the VC31 fully, by turning it anti-clockwise. Fully close the needle valve on the cryostat entry arm.

Connect the rotary pump to the rotary pump port, and switch it on. Make sure the valve on the pump inlet is fully open.

Check that the PTFE seal on the end of the transfer tube is clean and undamaged. There should be no grease on it.

Open the exhaust valve of the liquid helium dewar to release any pressure, keeping your hands and face away. Remove the plug in the transfer tube entry fitting. Slowly lower the dewar leg of the transfer tube into the liquid helium. Some liquid will be used to cool the leg, and the dewar exhaust must be open to allow this gas to escape. If you try to cool the leg too quickly a large amount of liquid will be wasted, and you could be burnt by the cold gas.

As soon as the dewar leg has been loaded into the liquid helium, push the other end into the entry arm of the cryostat until the knurled nut just touches the thread on the arm. Do not engage the thread yet. This allows liquid helium to bypass the cryostat, passing straight from the transfer tube into the entry arm and back into the exhaust, cooling the transfer tube quickly. Connect the 'from cryostat' fitting on the VC31 to the exhaust from the GFS600 as shown in Figure 2. This flow should increase gradually as the transfer tube cools. After a few minutes when the flow is about 1.5 litres per hour, engage the nut on the transfer tube on the thread on the cryostat arm and tighten it. This brings the PTFE seal into contact with its seat in the cryostat, forcing the helium to pass through the cryostat. If the flow does not reach 1.5 l/h after 20 minutes, the transfer tube may be blocked, or insufficient vacuum, or the needle valve may not be opening correctly. Refer to the transfer tube manual for further details.

If you have persistent problems with blockages in the transfer tube you may be able to reduce the risk by altering the procedure as follows. Before you start to lower the transfer tube leg into the storage dewar, connect the gas line from the VC31 to the cryostat end of the transfer tube using a short length of rubber tube. Use the GF3 pump and VC31 to draw helium gas through the transfer tube until it is cold and then, wearing thick gloves, quickly remove the gas line and insert the transfer tube into the cryostat arm. Engage the nut on the transfer tube on the thread on the cryostat arm and tighten it. Connect the gas line to the normal exhaust port and continue as described in the next paragraph.

Now that you have tightened the nut connecting the transfer tube to the cryostat, the flow rate will drop, because of the impedance of the warm pot in the cryostat. As this pot cools the flow will increase again, and after about 10 minutes it should be at least 1.5 litres per hour again. If not, there may be a blockage in the cryostat.

At this stage the heat exchanger will not show signs of cooling because all the flow of liquid passes out of the cryostat through the transfer tube. About ten minutes after you have started to cool down the cryostat you can start to cool the heat exchanger by slowly opening the needle valve on the cryostat entry arm (by approximately half a turn)

The sample space should cool steadily to about 2 K over about 90 minutes (assuming the ITC heater control is set to zero, or the SET temperature is well below 2 K). The exhaust gas flow through the transfer tube should still be at the maximum rate (2.5 litres/hour or more).

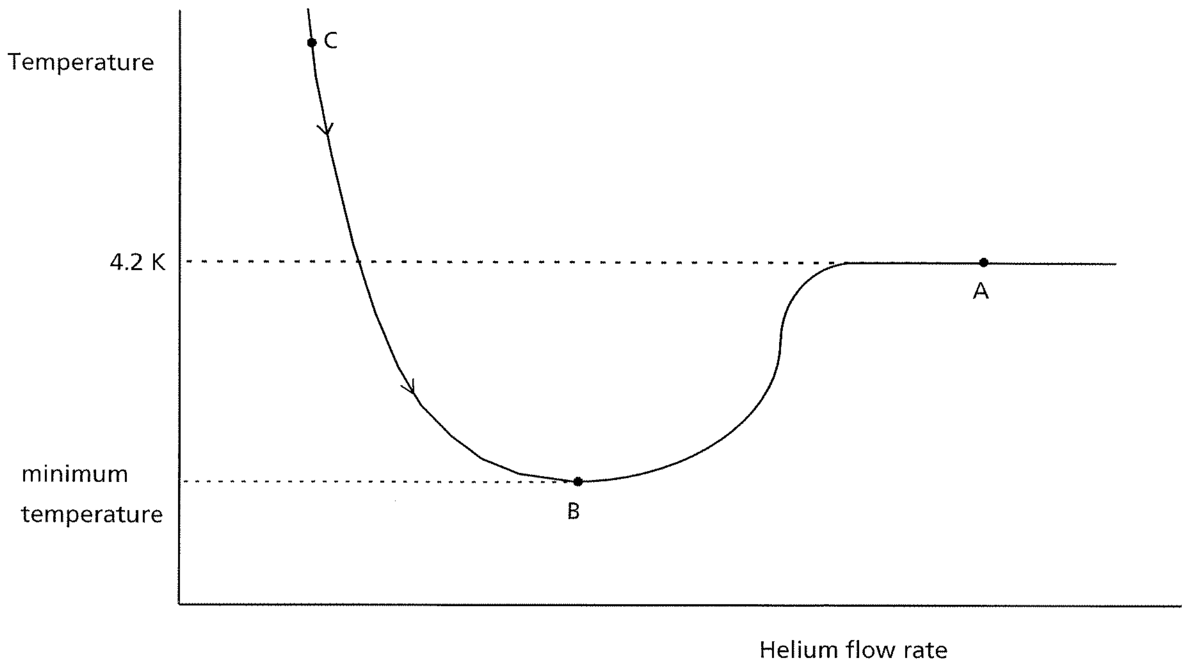

Figure 3 Temperature *vs* Helium flow rate, for a cryostat running in continuous flow conditions, zero heater voltage, standard entry arm

3.2. Operation below 4.2 K: version with low temperature entry arm

Temperatures lower than 4.2 K are achieved by lowering the pressure in the heat exchanger. Since the pumping speed of any pump is limited, this can only be achieved by limiting the rate at which helium is supplied to the heat exchanger, using the needle valve in the cryostat entry arm. The specifications or test results for the cryostat give the temperatures which can be achieved using the EPS25 rotary pump. The dependence of temperature on flow rate for a given pump is illustrated in Figure 3. It is important for continuous operation at low temperatures that the cryostat is not running in single shot mode, i.e. with a pool of excess liquid helium in the heat exchanger. To prevent this, you should use the following procedure.

Put the heater control and the gas flow control of the ITC temperature controller into MANUAL mode, with zero heater voltage. When the cryostat has reached 4.2 K (point A in Figure 3), close the needle valve on the entry arm. The temperature will probably fall immediately, because liquid helium in the heat exchanger is being boiled off. After at most a few minutes the liquid will have boiled away, and the temperature will start to rise.

At this point, open the needle valve on the entry arm about a quarter turn. The temperature should stabilise below about 20 K (at point C). Now open the needle valve in small increments, waiting for the temperature to stabilise after each change. As you do this, the temperature will fall, until you reach the base temperature of the system (point B).

If you want to run continuously with optimum efficiency you have to set the needle valve in the VC31 so that just enough liquid flows into the entry arm reservoir to replace the liquid that is being drawn into the cryostat. One way to do this is to close the needle valve on the VC31 until the cryostat has warmed to about 20 K, indicating that the entry arm reservoir is empty. Then slowly open the needle valve in the VC31, waiting for the temperature to stabilise after each change. As you do this, the temperature will fall, until you again reach the base temperature of the system.

Now select the desired SET temperature on the ITC, and switch the ITC heater control to AUTO.

3.3. *Temperature control above 4.2 K*

3.3.1. *Introduction*

You can control the temperature of the heat exchanger between 4.2 K and 500 K using a temperature controller. The flow of liquid helium and the heater power have to be adjusted to reach the required set point. The ITC^{502} or ITC^{503} temperature controller is used to control the heater power automatically, and adjusts the power to maintain the set temperature. These temperature controllers are three term controllers. The temperature control is optimised by setting the best values for:

- proportional band (P)
- integral action time (I)
- derivative action time (D)

The values given in the test results for the system are suitable to give good stability. If you want to improve the stability further you may be able to do this by adjusting the three terms slightly. The autotune facility on the ITC^{503} can be used to optimise these values, and the auto PID feature can be set up to allow the temperature controller to choose the best values for the three terms to suit the set temperature. The procedure for optimising the PID values and control theory are given in the ITC manual.

3.3.2. *Controlling at a 'set temperature'*

Select the channel on the temperature controller corresponding to the sensor which will be used to control the system, and ensure that the light on the heater control panel corresponds to the control sensor.

Set the required 'set temperature' by pressing and holding the SET button on the temperature controller, and using the RAISE/LOWER buttons to adjust the value shown on the main display. Set the PID values and the cryogen flow rate to those shown for the nearest temperature in the test results. Press the AUTO button once, and the temperature controller should adjust the heater output to warm the heat exchanger to the 'set temperature'.

You should then optimise the flow of liquid helium so that the heater output of the temperature controller is not too high. In general, the flow should be reduced until the steady heater output is at a suitable level. If you are using an Auto GFS system with a standard entry arm it will optimise the flow and heater voltage automatically, if you set the gas flow control to AUTO. As a guide, if you are optimising the flow manually the heater voltage should typically be as follows:

- 3 to 5 volts when the system is working in the region 4.2 K to 20 K
- 8 to 12 volts when it is working below 300 K
- greater than 8 volts when it is working above 300 K

If the cryostat has a standard entry arm, you adjust the needle valve on the transfer tube to optimise the flow of helium.

If you have a low temperature entry arm, the best procedure is as follows. Adjust the needle valve on the entry arm, with the VC31 valve fully open, until the heater voltage is in the desired range at the set temperature. Then gradually close the needle valve on the VC31 until the cryostat has warmed above its set temperature, indicating that the entry arm reservoir is empty. Then slowly open the needle valve in the VC31, waiting for the temperature to stabilise after each change. As you do this, the temperature will fall, until you again reach the set temperature.

3.3.3. Operation above room temperature

You can control the system at a 'set temperature' as described in section 3.3.2.

3.4. Warming up the system

Switch off the pump(s). If you do not need to warm the system quickly it may be left to warm up naturally. To speed the process, set a temperature of 300 K.

3.5. Changing samples

When the system is at room temperature the samples can be changed by simultaneously breaking the outer and inner vacuums and removing the system from the users apparatus

3.6. *Operating with liquid nitrogen*

Optistat[CF] cryostats can also be operated with liquid nitrogen instead of liquid helium. The basic operating procedure is the same as that for helium, but there are a few differences.

a) The flow gauge on the VC31 is calibrated for helium gas, so it will not give the correct flow reading for nitrogen gas. If the calibration is important, you could use a VC40 instead.

b) The viscosity of liquid nitrogen is higher than that of liquid helium so the flow rate through the cryostat is lower. This increases the cooldown time.

c) If you pump the liquid nitrogen to a pressure below 150 mbar you may freeze it and block the cryostat. A GF3 pump is unlikely to reduce the pressure sufficiently, but a rotary pump could.

d) It is more difficult to control the temperature of the sample, and specification is typically changed to $\pm$ 0.2 K. It is particularly difficult to control the temperature below 90 K, because liquid collects in the heat exchanger and boils intermittently.

e) Liquid nitrogen is not cold enough to cryopump air effectively, so it is more difficult to maintain a good vacuum in the transfer tube. It may be necessary to pump the OVC and transfer tube continuously because the warm surfaces outgas slightly. Surfaces cooled by liquid helium would freeze this gas and maintain the vacuum.

f) It is best to use the minimum flow possible to get good stability at low temperatures, (especially below 100 K). If you find that the temperature seems stable for a short time and then it suddenly becomes unstable, try to reduce the flow. Change the flow rate slowly, (typically 1% per minute), so that any liquid that has collected in the heat exchanger has time to boil away before you make another change.

g) When you find the optimum flow rate for base temperature this should be suitable for the whole temperature range. Increase it if you want to cool down more quickly, but as you approach base temperature reduce the flow again so that the cryostat is not filled with liquid.

h) If you are using an Auto GFS transfer tube it is best to run it in MANUAL mode. Since liquid nitrogen is much less expensive than liquid helium there is little advantage to be gained by reducing the consumption. When the system is in AUTO mode it may change the flow rate too rapidly, and good stability may never be achieved.

i) The PID settings on the temperature controller may be different from those given in the test results. Typically the P and I values should be increased slightly.