Operator's Handbook

# Microstat-HiResII

## (standard and pillared options)

Revision 4

Aug 08

UMC0029

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OXFORD INSTRUMENTS

# Contents



Calibration and test results appear after this manual.

# 1 Important information

**Warning**

Before you operate this equipment you must make sure that you are aware of the precautions necessary to ensure your own safety. We supply a separate booklet called *Safety Matters* with the system. Please read it carefully so that you fully understand the hazards you may encounter when using cryogens.

Oxford Instruments cannot accept responsibility for damage to the system caused by failure to observe the correct procedures laid down in this manual. The warranty may be affected if the system is misused, or the recommendations in this handbook are not followed.

## Important note - temperature and voltage limits

If you have bought a cryostat and temperature controller together from Oxford Instruments the temperature controller will have been set up in the factory:

- To prevent you from accidentally exceeding the maximum safe operating temperature of the cryostat
- To limit the maximum heater voltage to a safe level

If you are planning to use an existing temperature controller, or a power supply or controller made by another manufacturer, you should take the same precautions. The recommended values for the 'Heater Voltage Limit' and the 'Temperature Limit ($T_{HOT}$)' are given with the test results for the cryostat.

**Warning** **If you do not safeguard the system it is possible to cause serious damage.**

# 2 Description of the system

## 2.1 Overview

The Microstat-HiRes is a continuous flow liquid helium cryostat designed principally to allow a sample to be cooled to a low temperature and studied with an optical microscope. The pillar option allows the sample to be placed inside the bore of a magnet. The window arrangement allows the sample to be brought close to the objective lens of the microscope with the sample mounted in vacuum and cooled by conduction. The cryostat may be operated in any orientation.

The sample space and radiation shields are thermally insulated from the room temperature surroundings by the outer vacuum chamber (OVC). This space is pumped to a high vacuum before the cryostat is cooled down but it is protected against accidental build-up of high pressures by a pressure relief valve. The cryostat may be operated in any orientation.

Rotate the top window flange to bring it as close as required to the top surface of the sample. When the cryostat is evacuated this flange can still be rotated but some force will be required.

Up to two windows can be fitted to the Microstat. Each window is permanently bonded into the OVC flange (only one window is available for the pillar option).

## 2.2 Continuous flow cryostats

The Microstat-HiRes is a continuous flow liquid helium cryostat designed principally to allow a sample to be cooled to a low temperature and studied with an optical microscope.

Continuous flow cryostats do not have an internal reservoir to store a supply of cryogens. The liquid is supplied from a separate storage vessel through an insulated transfer tube. The transfer tube delivers the liquid helium to a heat exchanger close to the sample space. The gas returning from the heat exchanger then cools the radiation shield and flows out of the cryostat. A thermometer and heater are mounted on the heat exchanger, and these can be used with a temperature controller to balance the cooling power of the cryogen and to control the temperature of the sample.

Either liquid helium or liquid nitrogen can be used in this cryostat. Temperatures down to about 77 K can be reached using liquid nitrogen, but even at these temperatures better temperature stability can be achieved using liquid helium. If liquid helium is used, it is possible to maintain a temperature below 4.2 K continuously using the standard gas flow pump (GF4) and controller (VC31). Lower temperatures can be achieved using a larger pump such as the EPS 25 rotary pump. Figure 1 illustrates the overall system configuration when the cryostat is run using an LLT or GFS transfer tube. The system can also be run using a TTL transfer tube, in which case the pump is attached to a port provided for the purpose on the cryostat. Figure 1 shows a transfer tube with an auto needle valve, but a manual needle valve can also be used.

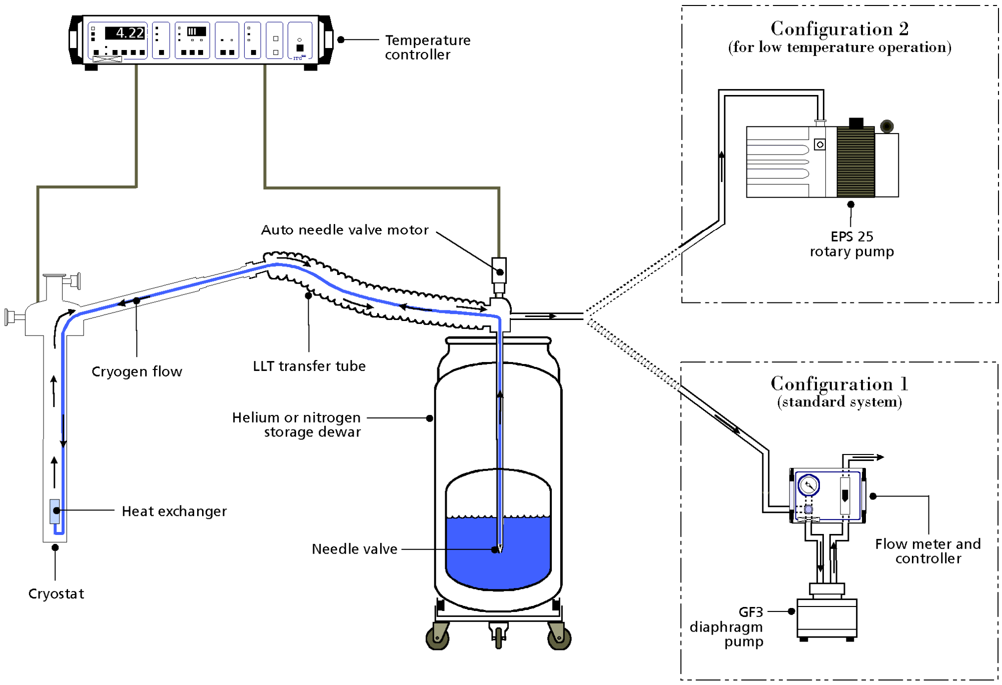


**Figure 1 Configurations for continuous flow cryostat systems**

## 2.3 The cryogen transfer tube

Two types of transfer tube are available for the Microstat-HiRes: TTL and LLT. (The GFS is an older design. It is identical in its use to the LLT, except that it is not recommended for operating in configuration 2 in Figure 1). They are described fully in separate manuals.

The TTL transfer tube is a low loss vacuum insulated transfer tube suitable for use with liquid helium or liquid nitrogen.

The LLT transfer tube is designed for ultra low loss performance. The cold exhaust gas from the cryostat flows along the tube, and the enthalpy of the gas is used to shield the flow of liquid from the room temperature surroundings. The LLT 600 and 700 are used for manual control, and the LLT 650 and 750 are automated versions, which allow the gas flow rate to be optimised automatically.

**TTL transfer tubes with external needle valve (black nut on the side of the dewar leg).** To close the needle valve, turn the black nut anti-clockwise (when viewed from above). To open the needle valve, turn the black nut in the opposite direction by no more than 6 turns. If you rotate the nut further, the thread will be disengaged and the factory setting will be lost.

**TTL transfer tubes with manual internal needle valve (black nut on top of the dewar leg), and**

**LLT or GFS transfer tubes with manual needle valve.** To close the needle valve, turn the handle clockwise. To open it fully, turn it by 6 turns anticlockwise.

**LLT, GFS or TTL transfer tubes with automatic needle valve.** To operate the needle valve manually, set the gas flow control on the ITC to MANUAL. To close the needle valve, set the gas flow to 0%. To open the needle valve, set the gas flow to 100%.

## 2.4 The gas flow pump and flow controller

The Oxford Instruments GF4 gas flow pump is used to promote the flow through the cryostat. It is an oil-free diaphragm pump.

The VC31 gas flow controller is used to control the flow of gas through the cryostat. It includes a flow meter (calibrated for helium gas) and a pressure gauge, so that the flow can be monitored.

## 2.5 Disposal and recycling

Before disposing of this equipment, it is important to check with the appropriate local organisations to obtain advice on local rules and regulations about disposal and recycling.

You **must** contact Oxford Instruments NanoScience Customer Support (giving full product details) before any disposal begins.

# 3 Unpacking and preparation

## 3.1 Unpacking the system

Carefully remove the cryostat and all the accessories from the packing case, and check the packing list to make sure that you have found all of the components. Examine the system to make sure that it has not been damaged since it left the factory. If you find any signs of damage please contact Oxford Instruments immediately.

To run this system you need the following components:

- Microstat-HiRes cryostat
- Liquid helium or liquid nitrogen storage dewar
- Cryogen transfer tube (TTL or LLT) and suitable adapter for storage dewar
- Polythene tube (7 mm inner diameter) for the gas exhaust
- High vacuum pumping system to evacuate the OVC.
- Oil-free diaphragm pump (such as the GF4): 3 – 500 K,
- OR rotary pump (such as the EPS 25) for operation below 3 K.
- Gas flow controller (VC31) and pumping lines
- Temperature controller (ITC 502 or ITC 503)

## 3.2 Preparing the system for operation

### 3.2.1 Evacuating the outer vacuum chamber (OVC)

The OVC has to be pumped to high vacuum to make sure that it gives the required thermal insulation. When the system is new, all of the materials inside the vacuum space are likely to outgas quickly, and this will affect the quality of the vacuum. This does not mean that the system is leaking, just that the new materials are being cleaned by the vacuum. The OVC should be pumped thoroughly before each cooldown, especially when the cryostat is new.

Connect the pumping system to the cryostat vacuum valve on the entry arm of the cryostat. We recommend that you use a diffusion pump or turbo-molecular pump, backed by a suitable pump. A diffusion pump should be fitted with a cold trap. Typically, you should pump the OVC until the pressure at the pump is $10^{-4}$ or $10^{-5}$ mbar. If the system is contaminated with water, the gas ballast facility on the rotary pump should be used.

When the system is new it is advisable to pump the system continuously if this is possible. Always use a cold trap in the pumping line if the system is used below ambient temperature at any time.

At high temperatures (i.e. > 100° C) internal components are effectively baked where adsorbed gas is released from internal surfaces. This additional gas load must be pumped away. If the cryostat is being swept in temperature it may show signs of sweating (i.e. the OVC may become cold to the touch or even start to condensate) if it is not on continuous pump. Again, this does not mean that the system is leaking, and the problem should reduce as the outgassing reduces with use.

To ensure the vacuum does improve it is important the when the system is not in use it is left either under vacuum or backfilled with dry nitrogen gas. If the vacuum space is left open to air for a long period of time, subsequent outgassing may begin to affect the vacuum integrity.

### 3.2.2 Evacuating the transfer tube

The transfer tube vacuum space has a separate evacuation valve similar to that on the cryostat, and the high vacuum pumping system can be connected to it directly.

### 3.2.3 Exhaust gas connections

If you are using a TTL transfer tube on the system, the exhaust gas is taken from the port on the cryostat entry arm. It does not flow back through the transfer tube.

If you have an LLT or GFS transfer tube, the exhaust port on the cryostat arm is closed, and the gas flows along the transfer tube to the exhaust port.

For standard operation (configuration 1 in Figure 1), a piece of polythene tube is used to connect the exhaust port on the cryostat or transfer tube to the VC31 gas flow controller. The other connections should be made as shown in Figure 3 or Figure 4 (depending whether you are using a TTL or LLT transfer tube) for continuous operation with the GF4 pump and VC31 controller. The exhaust line from the VC31 can either be connected to a helium recovery system or vented to the atmosphere.

For low temperature operation with the LLT transfer tube (configuration 2 in Figure 1), remove the “Christmas tree” nozzle clamped to the NW16 Klein flange on the transfer tube exhaust port. Attach a pumping line directly to this fitting, and attach the other end to the EPS 25 rotary pump. Make sure that an oil-mist filter is attached to the exhaust of the pump. The outlet of the oil-mist filter can either be connected to a helium recovery system or vented to the atmosphere.

### 3.2.4 Electrical connections to the temperature controller

| ITC socket (back panel) | Cryostat or transfer tube plug | Function |
|---|---|---|
| Sensor 1 | Heat Exchanger (cryostat) | Used to control and display the temperature of the heat exchanger. |
| Sensor 2 | Pillar sensor (cryostat) (Only available for pillar option) | Displays the temperature of the Cernox sensor in the copper sample pillar. |
| Auxiliary | Needle valve cable (transfer tube. Only used if there is an automatic needle valve) | Controls the rate of flow of liquid helium |

Check that the mains voltage selector on the temperature controller is correct for your local power supply, and connect it to the mains. Switch on the temperature controller, and press the SENSOR button until Sensor 1 LED is lit. The main display should then show the code for the sensor fitted to the heat exchanger of the cryostat. The correct code is shown in the test results sheet. The calibration in the temperature controller has been set up for the thermometer in the cryostat. If the code displayed is incorrect, please refer to the temperature controller manual.

### 3.2.5 Loading the sample

Warm the cryostat to room temperature if necessary, as described in section 4.5. Open the vacuum valve. Remove the top window flange by unscrewing it. Place the sample on the sample holder. A very thin layer of grease (such as Apiezon N grease) will improve thermal contact to the heat exchanger.

The sample holder can also be removed if required. To do this, first remove the top part of the copper radiation shield. Then remove the sample holder by undoing the four screws which hold it to the heat exchanger.

When replacing the sample holder or radiation shield, do up the screws tightly to ensure good thermal contact. Before re-installing the top window flange check the 'O' ring is clean, undamaged and lightly greased.

### 3.2.6 Making electrical connections to the sample: LX10 Additional Wiring

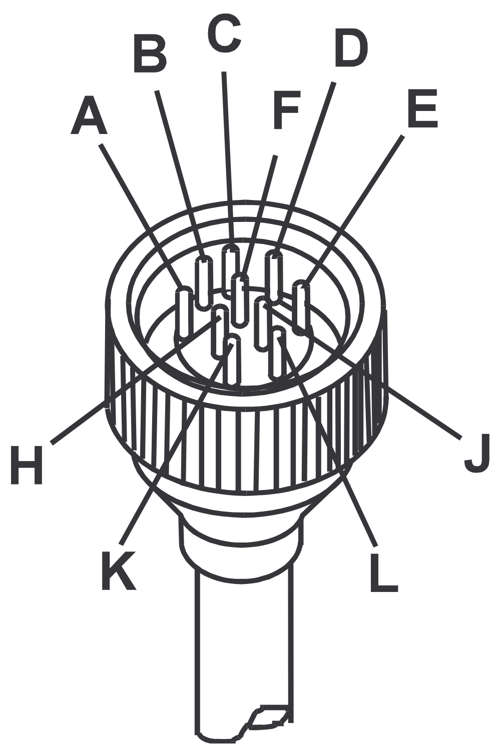


**Figure 2, Naming of pins in 10-pin plug**

This cryostat may be fitted with the "LX10" option. This consists of ten copper wires wired from a 10-pin plug on the outside of the cryostat to ten pins on a plastic plate fixed to the heat exchanger. To access these pins, remove the top window flange by unscrewing it, then remove the top part of the radiation shield by undoing the four screws which are now visible.

The connector is labelled "LX10". The pins in the plug are named A, B, C, D, E, F, H, J, K, and L. They are not labelled on the outside, but the corresponding contacts on the socket provided are labelled in tiny letters. The letters A and L should be engraved in the plastic ring to indicate the order of wiring.

Pins A and B are wired with 0.23 mm diameter wire. Pins C through to L are wired with 0.12 mm wire.

The wires to the sample should be thermally anchored to the heat exchanger or sample holder to make sure that too much heat is not introduced to the sample through the wiring.

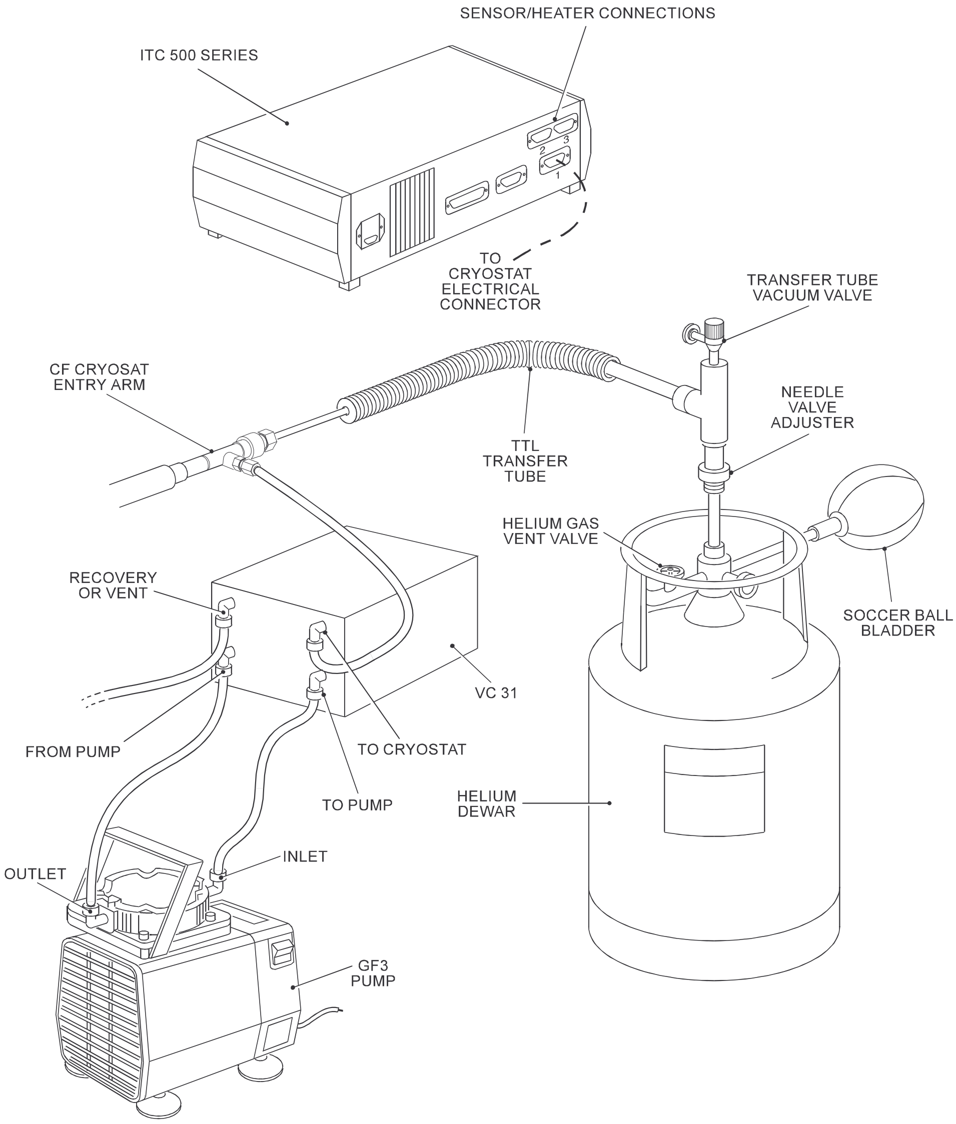


**Figure 3 Cryogen, exhaust gas and electrical connections (TTL transfer tube with external needle valve)**

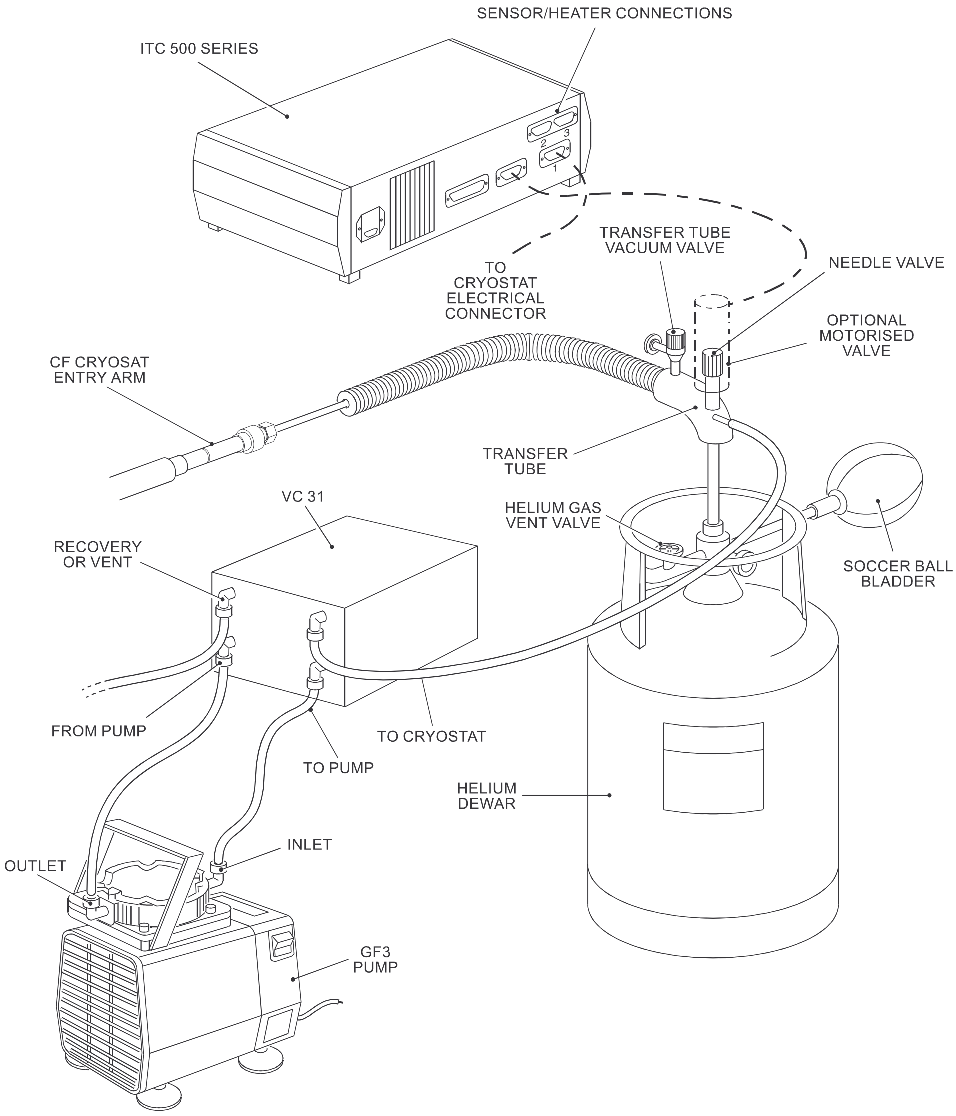


**Figure 4 Cryogen, exhaust gas and electrical connections (LLT transfer tubes)**

# 4 Running the system

The following procedure assumes that you are using liquid helium with the system. The Microstat-HiRes can also be used with liquid nitrogen, but some of the techniques are different. Please see section 4.6 for details. Ensure that the cryostat's OVC and the transfer tube have recently been pumped to high vacuum. Connect the system together as described in Section 3.2.

Set the 'set temperature' of the temperature controller to below 4.2 K, by pressing and holding the SET button, and using RAISE/LOWER to adjust the set point shown on the main display.

## 4.1 Cooldown using diaphragm pump

This set-up is illustrated in configuration 1 of Figure 1.

Make sure the needle valve on the transfer tube is fully open (see section 2.3).

Open the needle valve on the VC31 fully, by turning it anti-clockwise. Switch on the GF4 pump.

Check that the white PTFE seal near 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 the cold gas could burn you.

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 exhaust gas line from the VC31 to the cryostat entry arm or transfer tube, depending on whether you are using a TTL or LLT (or GFS) transfer tube. Watch the flow gauge on the VC31. 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 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 GF4 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 capillary tube in the cryostat. As this tube 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. Refer to section 7 for a description of the procedure to clear blockages.

The cryostat should now be cooling steadily, and the transfer tube and cryostat arm may contract by different amounts. The knurled nut on the cryostat arm should be tightened again occasionally, to make sure that it maintains the seal in the cryostat, so that the liquid helium does not by-pass the cryostat.

## 4.2 Cooldown using rotary pump

This set-up is illustrated in configuration 2 of Figure 1.

Make sure the needle valve on the transfer tube is fully open (see section 2.3).

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 the cold gas could burn you.

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 exhaust gas tube from the pump to the flange on the transfer tube and switch on the rotary pump. After a few minutes the exhaust tube should be cold, with condensation starting to form. 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 exhaust tube is not cold to the touch after about 20 minutes, the transfer tube may be blocked, 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 rotary pump to the cryostat end of the transfer tube using a short length of rubber tube. Use the pump to draw helium gas through the transfer tube until the exhaust tube 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 exhaust gas tube from the pump to the flange on the transfer tube 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 capillary tube in the cryostat. As this tube cools the flow will increase. After 20 minutes the exhaust tube should be cold again (assuming the ITC is set to a low temperature). If not, there may be a blockage in the cryostat. Refer to section 7 for a description of the procedure to clear blockages.

The cryostat should now be cooling steadily, and the transfer tube and cryostat arm may contract by different amounts. The knurled nut on the cryostat arm should be tightened again occasionally, to make sure that it maintains the seal in the cryostat, so that the liquid helium does not by-pass the cryostat.

## 4.3 Operation below 4.2 K

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, using the needle valve in the transfer tube. The specifications or test results for the cryostat give the temperatures which can be achieved using the GF4 diaphragm pump and using the EPS 25 rotary pump. The dependence of temperature on flow rate for a given pump is illustrated in Figure 5. 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 5), close the needle valve on the transfer tube. 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 about a quarter turn. The temperature should stabilise below about 20 K (at point C). Now open the needle valve in very 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).

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

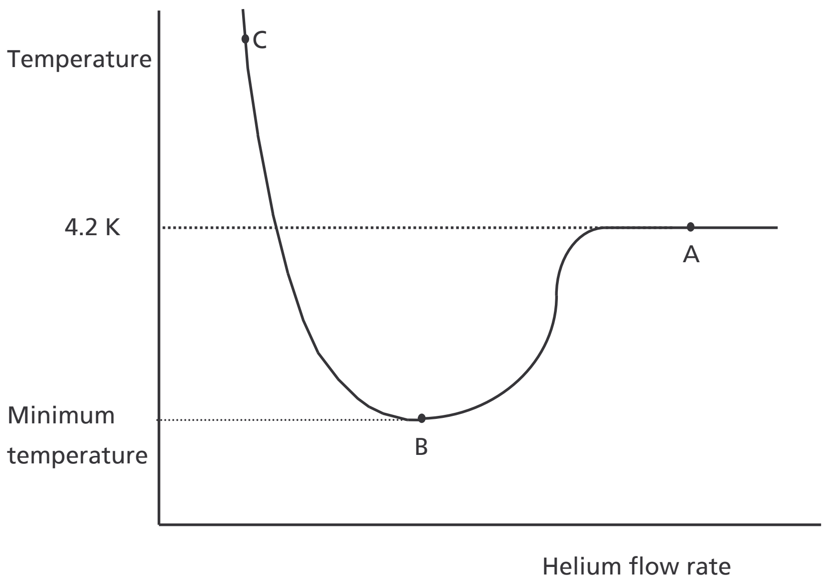


**Figure 5 Temperature *vs* Helium flow rate, for a cryostat running in continuous flow conditions, zero heater voltage**

# 4.4 Temperature control above 4.2 K

## 4.4.1 Introduction

You can control the temperature of the heat exchanger between 4.2 K and the specified maximum temperature of your cryostat using a temperature controller. The flow of liquid helium and the heater power have to be adjusted to reach the required set point. The ITC502 or ITC503 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 ITC503 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.

## 4.4.2 Controlling at a 'set temperature'

Check that the cryostat has been connected to the temperature controller as described in section 3.2. 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'.

It is not necessary to cool the cryostat to 4.2 K before you set the required 'set temperature'. If the temperature controller is set to the required temperature at the beginning of the cooldown, the cryostat should cool to the set temperature and the temperature controller should then hold it at this point.

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 LLT system it will optimise the flow and heater voltage automatically for you. 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 in the region 20 – 300 K

## 4.5 Warming up the system

Switch off the gas flow pump. After a few seconds the pressure in the helium flow circuit will rise to approximately the pressure of the storage dewar, and the transfer tube can be removed from the cryostat. Immediately fit the special pressure relief valve (supplied with the system) into the cryostat so that it is not contaminated with ice (condensed from the air). This could block the transfer tube next time it is cooled down.

If you do not need to warm the system quickly it may be left to warm up naturally. If you want to warm it more quickly allow a small volume of dry nitrogen gas into the OVC to break the vacuum. You can use the temperature controller to help warm the cryostat.

## 4.6 Operating with liquid nitrogen

The cryostat 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 VC41 instead.

b) The cooldown time is greater with nitrogen.

c) If you pump the liquid nitrogen to a pressure below 150 mbar you may freeze it and block the cryostat. The pressure can be regulated using the needle valve located on the VC series of gas controllers (if purchased). If you are not using an Oxford Instruments gas controller then it will be necessary to use a valve on the pumping line to maintain a pressure above 150 mbar in the cryostat.

d) It is more difficult to control the temperature of the sample, and specification is typically changed to ± 0.2 K. It is particularly difficult to control the temperature below 90 K, because liquid collects in the sample space 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 OVC and transfer tube. It may be necessary to pump the OVC and transfer tube continuously because the warm surfaces outgas slightly.

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 sample space heat exchanger has time to boil away before you make another change.

g) When you find the optimum flow rate for 77.3 K this should be suitable for the whole temperature range. Increase it if you want to cool down more quickly, but as you approach 77.3 K reduce the flow again so that the cryostat is not filled with liquid.

h) If you are using an LLT or GFS transfer tube with an automatic needle valve it is best to run it with the gas flow control 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 gas flow control 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.

# 5 Maintenance

Check the 'O' rings on the top and bottom window flanges, and the one in the transfer tube entry arm for cuts or abrasions.  If you find a damaged ring, replace it.

# 6 Electrical connections

The standard cryostat is fitted with a ten-pin plug on the side of the cryostat. The plug is held in place by the black nut. Do not remove it unless you need to gain access to the wiring, as this will allow air into the vacuum chamber.

As standard, the heat exchanger is fitted with a rhodium iron temperature sensor (set up for four-wire measurement) and a heater. Detailed information about the temperature sensor is given at the end of this manual.

| Pin | Function | |
|---|---|---|
| A | ) | Heat exchanger heater |
| B | ) | (Watlow Firerod cartridge heater) |
| C | ) V+ | |
| D | ) V- | Heat exchanger sensor |
| E | ) I+ | |
| F | ) I- | |

## 6.1 Checking the wiring

A resistance meter can be used to check the wiring of the cryostat. You should expect to measure the following values. These readings may be affected if the cryostat is damp or if your fingers are in contact with one or more of the pins.

| **Pins of heat exchanger plug** | **Expected resistance.** Measured values are in the wiring sheet appended to this manual. |
|---|---|
| A to B | 42 Ω approx. |
| C to D | 72 Ω approx. |
| C to E | 42 Ω approx |
| C to F | 72 Ω approx. |
| D to F | 42 Ω approx. |
| A to C | > 1 MΩ |
| A to ground | > 1 MΩ |
| C to ground | > 1 MΩ |

## 6.2 Pillar option: Cernox Sensor

An additional Cernox sensor may have been positioned close to the end of the copper pillar if this option was been chosen when purchasing the cryostat. The wiring configuration for this is given below:

| **Pin** | **Function** | |
|---|---|---|
| A | ) | No Connection |
| B | ) | No Connection |
| C | ) V- | |
| D | ) V+ | Heat exchanger sensor |
| E | ) I+ | |
| F | ) I- | |

A wiring check should result in the following approximate values:

| **Pins** | **Expected resistance.** Measured values are in the wiring sheet appended to this manual. |
|---|---|
| C to D | 113 Ω approx. |
| C to E | 113 Ω approx |
| C to F | 58 Ω approx. |
| D to F | 58 Ω approx. |
| | |
| C to ground | > 1 MΩ |

# 7 Fault finding

The following table shows the most common faults. Refer also to the fault-finding table in the transfer tube manual.

| Symptom | Diagnosis and suggestions |
|---|---|
| Cryostat outer vacuum chamber (OVC) cannot be pumped to high vacuum<br><br>Or<br><br>Water condenses on the cryostat body when it is cold | Check the OVC for leaks. In particular check:<br>• cryostat top and bottom plate O-rings<br><br>If there is no leak there may be too much moisture in the OVC and it should be pumped with a rotary pump, with the gas ballast valve open. |
| Cryostat will not cool down | Check whether there is any flow of gas through the system, using the gauge on the VC31. If not see the transfer tube manual. |
| Poor temperature stability | Check that the PID settings on the temperature controller and the cryogen flow rate are as suggested in the test results. |
| Cryostat cannot be warmed up.<br><br>or<br><br>Heater not working | Check that the 'set temperature' is higher than the present sample temperature, or switch the heater on manually.<br><br>Check that the high temperature limit of the temperature controller has not been exceeded, (as indicated by the message "Hot 1" on the display).<br><br>Check that the heater voltage limit on the temperature controller is high enough.<br><br>Check that the heater is not open circuit by checking from pin A to pin B. If so the wiring will have to be repaired. |

| Symptom | Diagnosis and suggestions |
|---|---|
| Cryostat will not reach specified minimum temperature | Check that the heater is switched off.<br><br>Check that the flow rate is high enough, and that there is sufficient liquid in the storage dewar. If the flow is high the liquid flow may be by-passing the cryostat. Check that the transfer tube nut is tight enough, and if so check that the PTFE seal has not been damaged. (See sections 3.1 and 3.2 for more information.)<br><br>Check the connections to the thermometer and make sure that it is working properly and in good thermal contact with the cryogen flow.<br><br>Check that you have not added too much heavy wiring to the sample holder, introducing a high heat load.<br><br>Check the quality of the vacuum in the OVC.<br><br>Check the vacuum in the transfer tube.<br><br>Check the cryostat for mechanical damage. Warm it to room temperature and remove the OVC to check whether the radiation shield touches the sample holder or OVC.<br><br>Check that the radiation shield has been fitted correctly.<br><br>Check that the sample or sample holder are not touching the radiation shield or OVC. |
| Sensor not reading correctly | Check the wiring. See section 6.1and 6.2. |