Operator's Handbook

OptistatCF

Static Exchange Gas Continuous Flow Cryostat

Revision 3

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Calibration and test results appear at the end of this manual.

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1 Important Information

1.1 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.

Caution: 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.

1.2 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.

Caution: If you do not safeguard the system it is possible to cause serious damage.

1.3 Important Note

This manual is part of the product that you have bought. Please keep it for the whole life of the product and make sure that you incorporate any amendments which might be sent to you. If you sell or give away the product to someone else, please give them the manual too.

1.4 Important Health and Safety Notice

Important Health and Safety Notice

When returning components for service or repair it is essential that the item is shipped together with a signed declaration that the product has not been exposed to any hazardous contamination or that appropriate decontamination procedures have been carried out so that the product is safe to handle.

1.5 Conventions used in this manual

The following conventions have been followed in this manual:

Danger: **Indicates that the hazard may cause death or severe injury if the instructions are not followed carefully.**

Warning: **Indicates that the hazard may cause injury.**

Caution: Indicates that the hazard may cause damage to equipment.

Note: Something that needs to be brought to the customer's attention.

Tip: Indicates a helpful hint that may be of use to the customer.

1.6 Disposal and recycling instructions

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.

2 Description of the system

2.1 The Cryostat

Figure 1 is a schematic diagram of the OptistatCF cryostat, showing most of the interesting features.

The sample is mounted on a removable sample holder, and positioned inside the window block. Up to five windows can be fitted to give optical access to the sample space. The sample can be rotated.

The sample space and radiation shields are thermally isolated 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.

A range of window materials is available, covering most of the electromagnetic spectrum. The inner windows are sealed into the window block by an indium seal. The techniques needed to replace them are described in section 6.3

The 500 K high temperature option of the OptistatCF has a stainless steel window block. (If you look through the windows you easily tell whether this block is copper or stainless steel). This block will accept

- Copper gasket sealed windows with a maximum temperature of 500 K
- Indium sealed windows with a maximum temperature 320 K

If you intend to run the system at temperatures above 320 K all five window ports must be fitted with copper gasket seals, not indium seals.

2.1.1 Version with low temperature entry arm

Figure 1 also shows the low temperature entry arm ("alternative entry arm for LT version"). This version also has a rotary pump port, which is the only exhaust outlet for the heat exchanger.

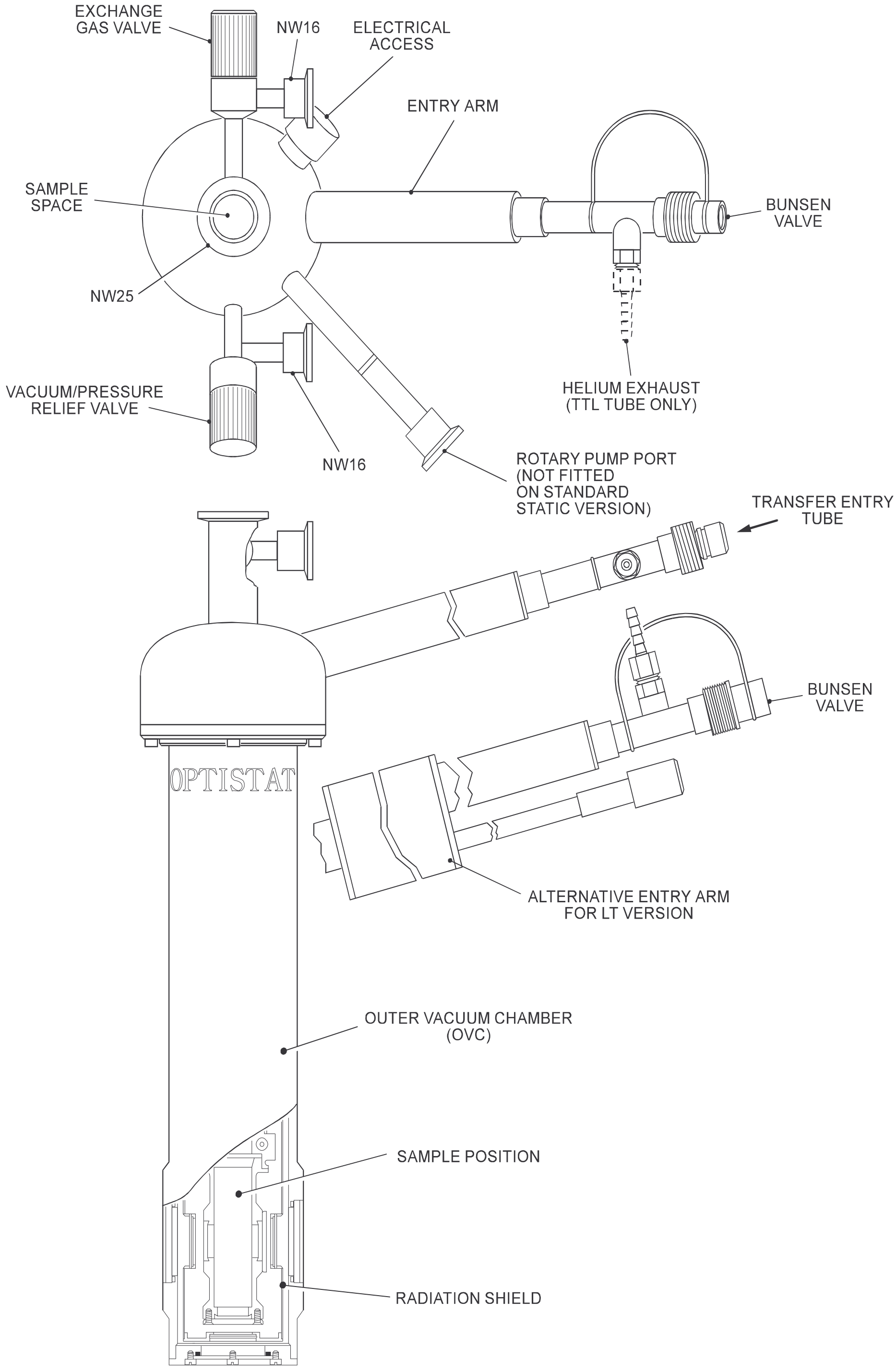

Figure 1 Schematic diagram of OptistatCF cryostat

2.2 The cryogen transfer tube

Two types of transfer tube are available for the OptistatCF: TTL and LLT. (The GFS is an older design. It is identical in its use to the LLT, except that you do not get a large improvement in performance by using configuration 2 in Figure 2). 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.

Caution: If you are using a TTL200 transfer tube, make sure that you do not unscrew the dewar leg completely, as this would change the original factory settings. If you do accidentally remove this leg, refer to the transfer tube manual, which describes the procedure for resetting it.

The LLT transfer tube is designed for ultra low loss performance. The cold exhaust gas from the cryostat flows along the tube, shielding the incoming liquid from room temperature thermal radiation. The LLT600 and 700 are used for manual control and the LLT650 and 750 are automated versions, which allow the gas flow rate to be optimised automatically using the ITC temperature controller.

TTL transfer tubes. 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 six turns. If you rotate the nut further, the thread will be disengaged and the factory setting will be lost.

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 six turns anticlockwise.

LLT or GFS 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%.

2.3 The gas flow pump(s) and flow controller

See section 2.1 for the components required for your system.

The Oxford Instruments GF4 gas flow pump is used to promote the flow through the cryostat. It is an oil free diaphragm pump with a nominal displacement of 42 litres per minute. The air leak rate is guaranteed to be less than 10 cm^3/minute.

The EPS25 is an oil-sealed rotary vane pump. It should be equipped with an oil mist filter on the exhaust, and a valve on the inlet. Whenever there is a large flow rate of air or helium through the pump, there is a risk that oil mist will escape through the oil mist filter. If this happens, check that the system is set up correctly, so that no air is being let in. If necessary partially close the valve to reduce the pumping speed, but note that the valve must be fully open to achieve the lowest possible temperature.

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.

2.4 Operating principles

A continuous flow cryostat does not have an internal reservoir to store a supply of cryogen. The liquid is supplied from a separate storage vessel through an insulated transfer tube. It flows through a heat exchanger around the sample space, and out of the cryostat to the pump. A thermometer and heater are mounted on the heat exchanger, and these can be used with a temperature controller to control the temperature of the heat exchanger. The sample is mounted in a separate space, which is filled with exchange gas. This gas gives good thermal contact between the heat exchanger and the sample. The flow of liquid which cools the cryostat does not come into direct contact with the sample.

In a static exchange gas system you can adjust the atmosphere around the sample to suit the experimental requirements. It is more difficult to accidentally block the cryostat with frost while changing the sample.

If the pressure in the sample space is kept low enough it is impossible for liquid to condense around the sample (where it may interfere with optical measurements). In addition, if you are doing high voltage experiments you may be able to reduce the risk of an electrical discharge by choosing a suitable sample space pressure.

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.

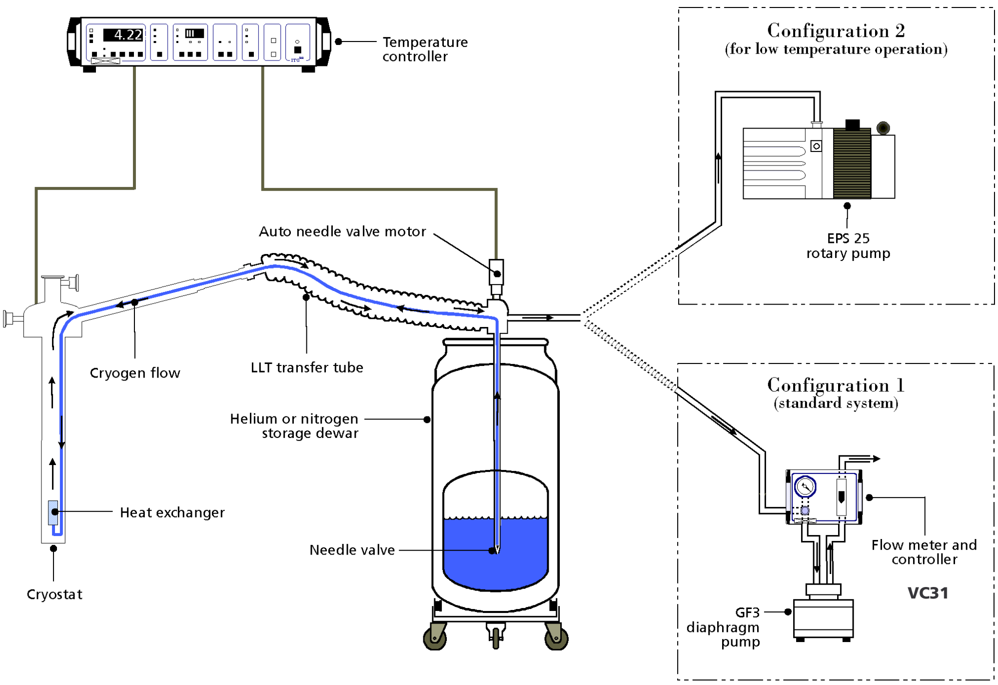

Figure 2 System Configurations (version with standard entry arm)

2.5 System configuration: version with standard entry arm

If liquid helium is used, it is possible to maintain a temperature slightly 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 2 illustrates the possible system configurations 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.

2.6 System configuration: low temperature entry arm version

The liquid is supplied to a small reservoir in the entry arm of the cryostat. The transfer tube and the VC31 are connected as shown in configuration 1 of Figure 2. The system can also be run using a TTL transfer tube, in which case the GF4 pump is attached to a port provided for the purpose on the cryostat. Figure 2 shows a transfer tube with an auto needle valve, but a manual needle valve would normally be used, as automatic gas flow control is not possible with TTL transfer tubes).

The valve on the VC31 is used to set the flow rate so that there is always a sufficient supply of liquid helium for the cryostat. The needle valve in the entry arm is then used to set the flow rate to the heat exchanger. The heat exchanger is pumped by a rotary pump (which typically has a displacement of 25 m^3/h). This pump reduces the helium vapour pressure to cool it to temperatures as low as 1.6 K continuously.

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:

- OptistatCF cryostat
- Liquid helium or liquid nitrogen storage dewar
- Cryogen transfer tube (TTL , LLT or GFS)
- Polythene tube (10 mm outer diameter) for the gas exhaust
- High vacuum pumping system to evacuate the OVC
- Bladder (usually a soccer ball bladder), with an adaptor nozzle for fitting it onto an NW16 flange (Klein flange)
- Source of exchange gas. This will normally be helium even if liquid nitrogen is used to cool the cryostat, as helium has better thermal conductivity.
- Fitting for connecting bladder to the source of exchange gas. The Oxford Instruments SV12 is suitable if a liquid helium dewar is used.
- Temperature controller (ITC502 or ITC503) with cryostat cable

Three configurations are possible:

Version with standard entry arm, used with diaphragm pump (configuration 1 in Figure 2.)

This requires:

- Diaphragm pump, such as the GF4
- Gas flow controller (VC31)

Version with standard entry arm, used with rotary pump to obtain lower temperatures (configuration 2 in Figure 2). This requires:

- Rotary pump, such as the EPS25. The EPS25 is supplied with a valve and pumping line. A T-piece with a vacuum gauge is optional.

Version with low temperature entry arm. This requires

- Diaphragm pump, such as the GF4, **and**
- Rotary pump, such as the EPS 25. The EPS25 is supplied with a valve and pumping line. A T-piece with a vacuum gauge is optional.

4 Preparing the system for operation

Choose a suitable position to operate the cryostat safely, and if necessary arrange for it to be supported so that it cannot accidentally fall. Use Figure 1 to help you to locate the services and controls on the top of the cryostat.

4.1.1 Evacuating the OVC

The OVC has to be pumped to high vacuum to make sure that it gives the required thermal isolation. When the system is new, all 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 vacuum valve on the top plate of the cryostat. We recommend that you use a diffusion pump or turbo-molecular pump, backed by a rotary pump, and fitted with a cold trap which helps the system to pump water vapour. Typically, you should pump the OVC until the pressure at the pump is 10^{-4} or 10^{-5} mbar. If the system is badly contaminated with water vapour, the gas ballast facility on the rotary pump should be used.

4.1.2 Evacuating the transfer tube

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

4.1.3 Exhaust gas connections: standard entry arm version

If you are using a TTL transfer tube, attach the adaptor nozzle to the helium exhaust on the cryostat entry arm. If you are using an LLT or GFS transfer tube, keep this port closed.

For standard operation (configuration 1 in Figure 2) connections should be made using the polythene tube as shown in Figure 3 or Figure 4 (depending whether you are using a TTL or LLT transfer tube), but do not connect the tube to the cryostat entry arm or transfer tube at this stage. 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 2), remove the adaptor nozzle clamped to the NW16 fitting (Klein flange) on the transfer tube exhaust port. Attach a pumping line to the rotary pump, with a valve connected between the pumping line and the pump inlet. A T-piece with a vacuum gauge is optional. 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.

4.1.4 Exhaust gas connections: low temperature entry arm version

The rotary pump is connected to the rotary pump port on the cryostat using the pumping line provided with the EPS 25, or another pumping line of at least 25 mm internal diameter. A valve should be connected between the pumping line and the pump inlet. A T-piece with a vacuum gauge is optional. 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.

If you are using a TTL transfer tube, attach the adaptor nozzle to the helium exhaust on the cryostat entry arm. If you are using an LLT or GFS transfer tube, keep this port closed. Connections should be made using the polythene tube as shown in Figure 3 or Figure 4 (depending whether you are using a TTL or LLT transfer tube), but do not connect the tube to the cryostat entry arm or transfer tube at this stage. The exhaust line from the VC31 can either be connected to a helium recovery system or vented to the atmosphere.

4.1.5 Electrical connections to the temperature controller

The ITC temperature controller should be connected to the cryostat as shown in Figure 3 or Figure 4. The cable should usually be connected to the "sensor 1" socket on the temperature controller. 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. While the button is held pressed the main display should then show the code for the type of temperature sensor fitted to the cryostat. The correct code is shown in the test results sheet. The calibration in the temperature controller has been set up for the temperature sensor in the cryostat. If the code displayed is incorrect, please refer to the temperature controller manual.

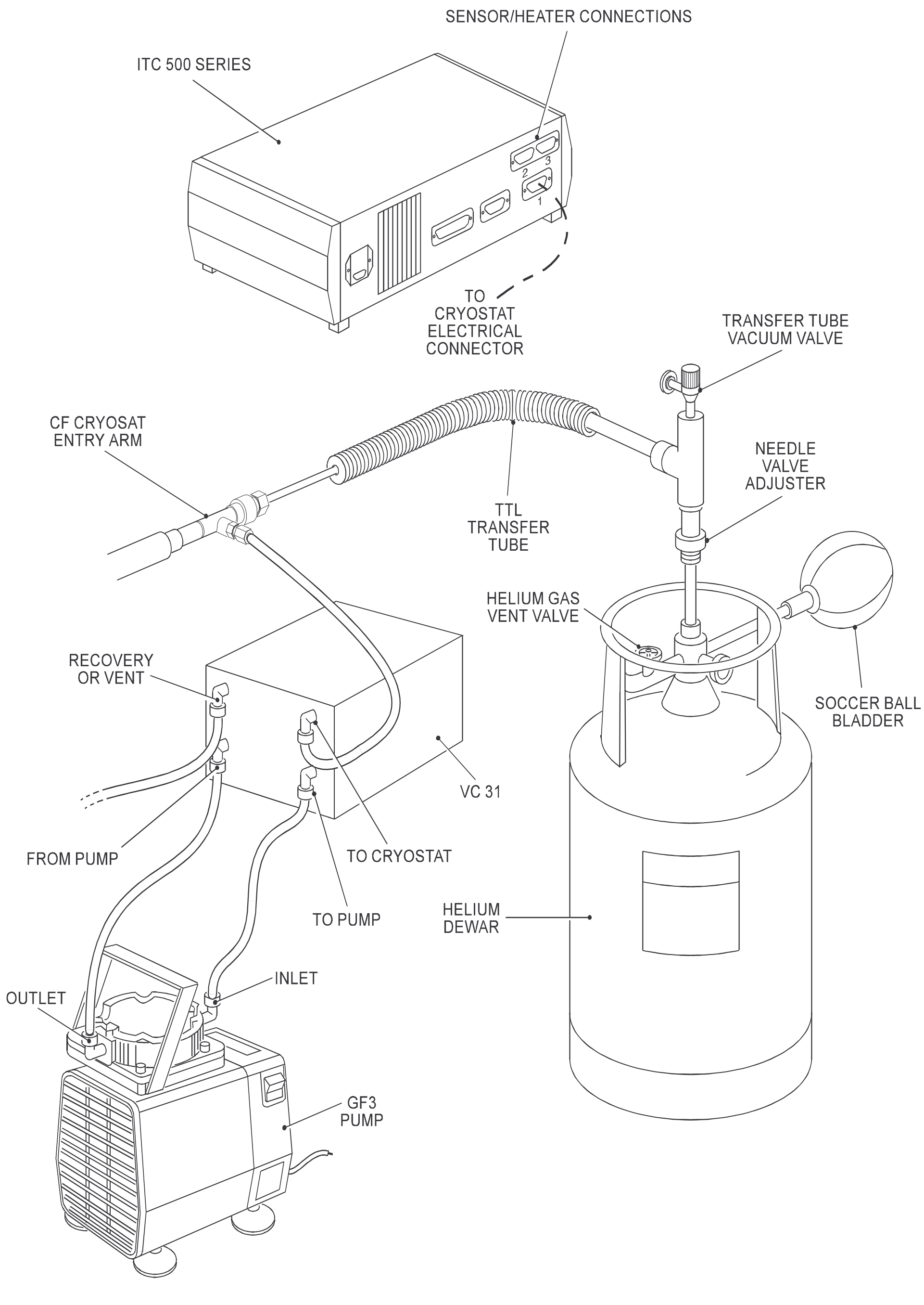

Figure 3 Cryogen, exhaust gas and electrical connections (version with standard entry arm, using a TTL transfer tube)

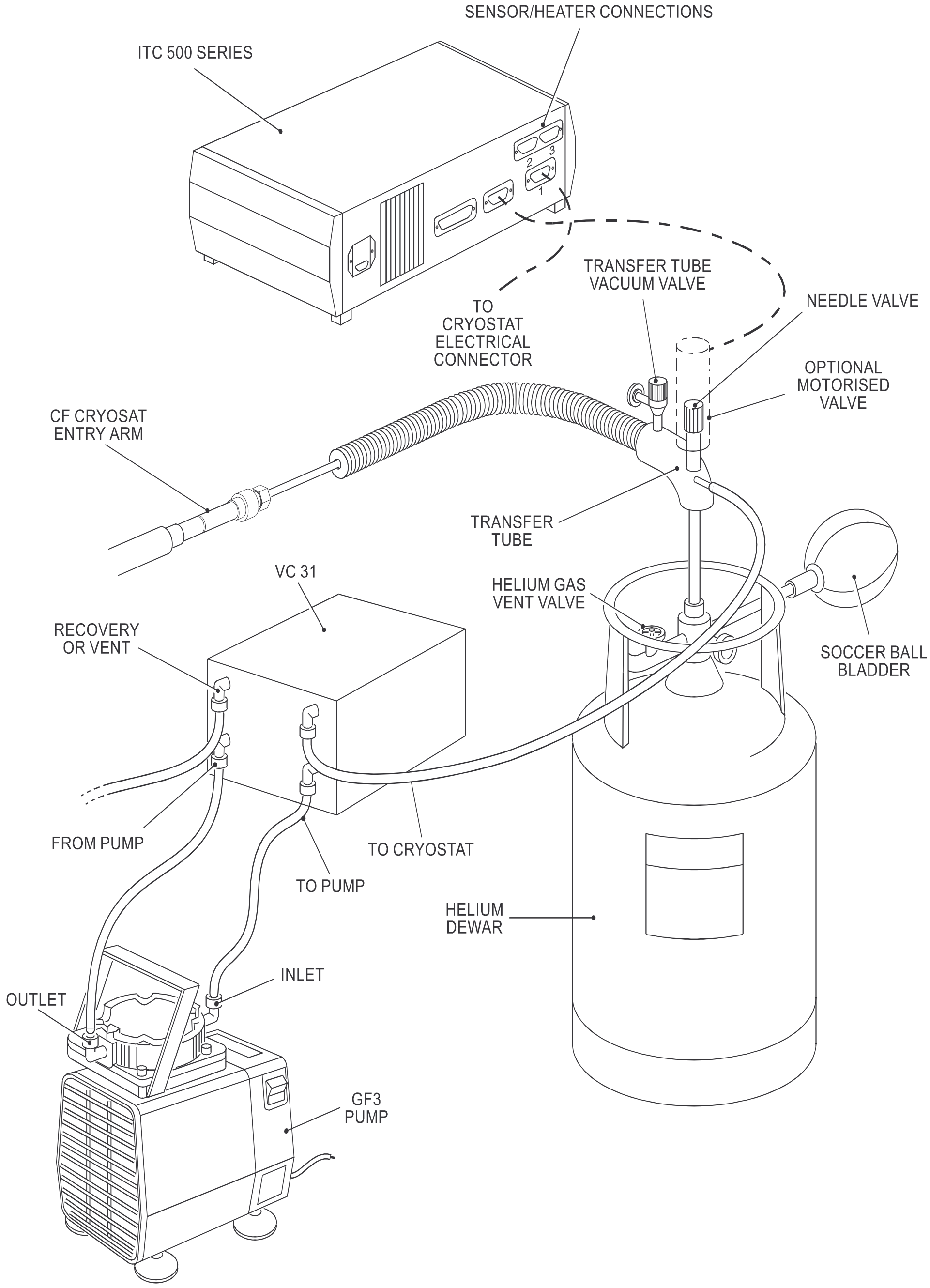

Figure 4 Cryogen, exhaust gas and electrical connections

(version with standard entry arm, using an LLT or GFS transfer tube)

5 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. OptistatCF cryostats can also be used with liquid nitrogen, but some of the techniques are different. Please see section 5.10 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 4.

Switch on the ITC and set the temperature display to show the sensor fitted to the heat exchanger. If you plan to run it above 4.2 K, you can set the heater control to AUTO, select the correct sensor for heater control, and select the desired temperature by pressing and holding the SET button, and using RAISE/LOWER to adjust the set point shown on the main display. If you plan to run it below 4.2 K, at this stage leave the heater control and the gas flow control in MANUAL mode, with the heater voltage set to zero.

5.1 Loading the sample and exchange gas

Put in the sample holder or fit the blank which is supplied with the system. Fill the sample space with your chosen exchange gas. The instructions below assume this is helium which is initially at atmospheric pressure.

Fill the bladder with helium. The liquid helium supply dewar is suitable for this purpose. You will need a suitable fitting for the dewar, such as the SV12, which has a valve to prevent the dewar depressurising suddenly when the bladder is removed. To pressurise the dewar slightly, squeeze and release the bladder a few times. This introduces warm helium into the dewar, accelerating the boil-off.

Evacuate the sample space with a vacuum pump connected to the exchange gas valve. Close the valve and attach the bladder using the adaptor nozzle, allowing some of the gas to escape to flush air out of the connection as you make it. Then open the valve to let helium gas into the sample space. Leave the valve open and the bladder connected. This helps you to monitor the pressure in the sample space. If you want to make sure that no liquid is condensed in the sample space close the exchange gas valve when the cryostat reaches about 20 K. This also helps to reduce the risk of allowing air into the sample space if there is a leak in the bladder. When you warm the cryostat up, open the exchange gas valve again to allow the exchange gas to expand into the bladder. The sample space is protected against dangerous pressure build up by a pressure relief valve, but it is not good practice to rely on it.

If you use a rotary pump, it is sufficient to pump once. If you use a diaphragm pump, such as the GF4, some air will be left after a single pump, so if it is essential that very little air is left in the sample space, you should pump and flush with helium two or three times.

It is most convenient to use a separate pump, but if necessary you can disconnect the main gas flow pump while you evacuate the sample space.

The procedure for changing samples is given in section 5.8.

5.2 Cooldown: version with standard entry arm, using diaphragm pump

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

Make sure the needle valve on the transfer tube is fully open. (See section 2.2.)

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

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 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 8 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.

5.3 Cooldown: version with standard entry arm, using rotary pump

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

Make sure the needle valve on the transfer tube is fully open (see section 2.2).

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 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 8 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.

5.4 Cooldown: version with low temperature entry arm

Make sure the needle valve on the transfer tube is fully open. (See section 2.2.) 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 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 8 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.

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.

The sample space should cool steadily to about 2 K over about 15 to 20 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).

5.5 Operation below 4.2 K: version with standard 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, 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 (increase gas flow by 35% if you have an automatic needle valve). 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).

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

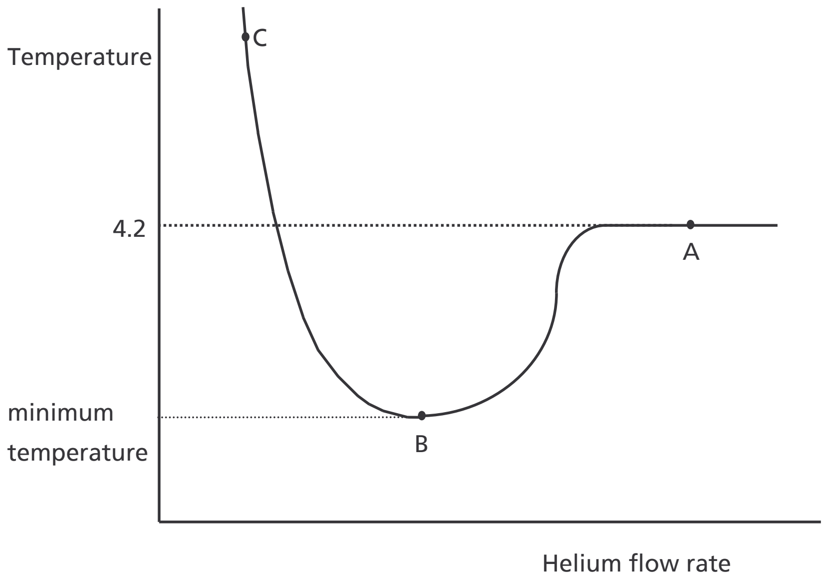

Figure 5 Temperature *vs* Helium flow rate, for a cryostat running in continuous flow conditions, zero heater voltage, standard entry arm

5.6 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 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 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.

5.7 Temperature control above 4.2 K

5.7.1 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 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.

5.7.2 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 LLT or 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.

5.7.3 Operation above room temperature

Before you set a temperature above room temperature, check that your system is suitable for this temperature range.

You may need to pump the OVC continuously to maintain the required high vacuum. You can now control the system at a 'set temperature' as described in section 5.7.2.

5.8 Changing samples

The sample holder can be removed and replaced while the cryostat is at any temperature above 4.2 K. The pump(s) can be on or switched off when samples are changed. However, remember that your sample may be subjected to thermal shocks if it is warmed or cooled too quickly. It will also become covered with ice if it is very cold when it is removed from the sample space.

Remove the NW25 clamp and lift the sample rod out of the sample space. When the sample space is opened, air and moisture may be allowed in, and these could fog the windows and contaminate the sample space. The best way to prevent this is to connect a supply of (or bladder of) dry helium gas to the sample space valve. A continuous flow of gas should be used to purge the sample space while it is open. Fit a blank flange to the top of the sample space as soon as possible.

5.9 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. If you want to warm it even more quickly allow a small volume of dry nitrogen gas into the OVC to break the vacuum. Leave a bladder connected to the exchange gas valve and leave the valve open. This ensures that the exchange gas can expand safely as it warms up. Never allow helium gas into the OVC as it is difficult to pump it out again. (Tip: do not use a bladder on the OVC that has previously been used with helium.)

5.10 Operating with liquid nitrogen

OptistatCF 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 VC41 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 GF4 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 ± 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 OVC and 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 LLT or 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.

6 Maintenance

If you carry out a few simple procedures your cryostat will give you years of reliable operation.

6.1 Rubber 'O' rings

Whenever you remove part of the cryostat or if you suspect that there is a leak on the system check the 'O' rings on the sample space, transfer tube entry arm and OVC for cuts or abrasions. If you find a damaged ring, replace it.

6.2 Removing the OVC and radiation shield

The OVC and radiation shield only have to be removed if you want to:

- Change the windows
- Modify or repair the wiring
- Repair mechanical damage

Warm the cryostat to room temperature and then open the OVC and sample space valves to allow air into the system. If there is any chance that the cryostat is not completely warm, use dry nitrogen gas instead of air. Refer to Figure 1. Remove the four screws which hold the OVC to the cryostat top plate, and carefully remove the OVC. You will then be able to see the small feed capillary tube which supplies liquid from the entry arm to the heat exchanger. It should pass through the slot in the radiation shield thermal link and it is important that it does not touch the shield or any of the other tubes. The slightly larger tube takes the exhaust gas from the radiation shield thermal link back to the entry arm

The radiation shield is fixed to the cryostat by four screws. These should be removed, if you need to take off the radiation shield.

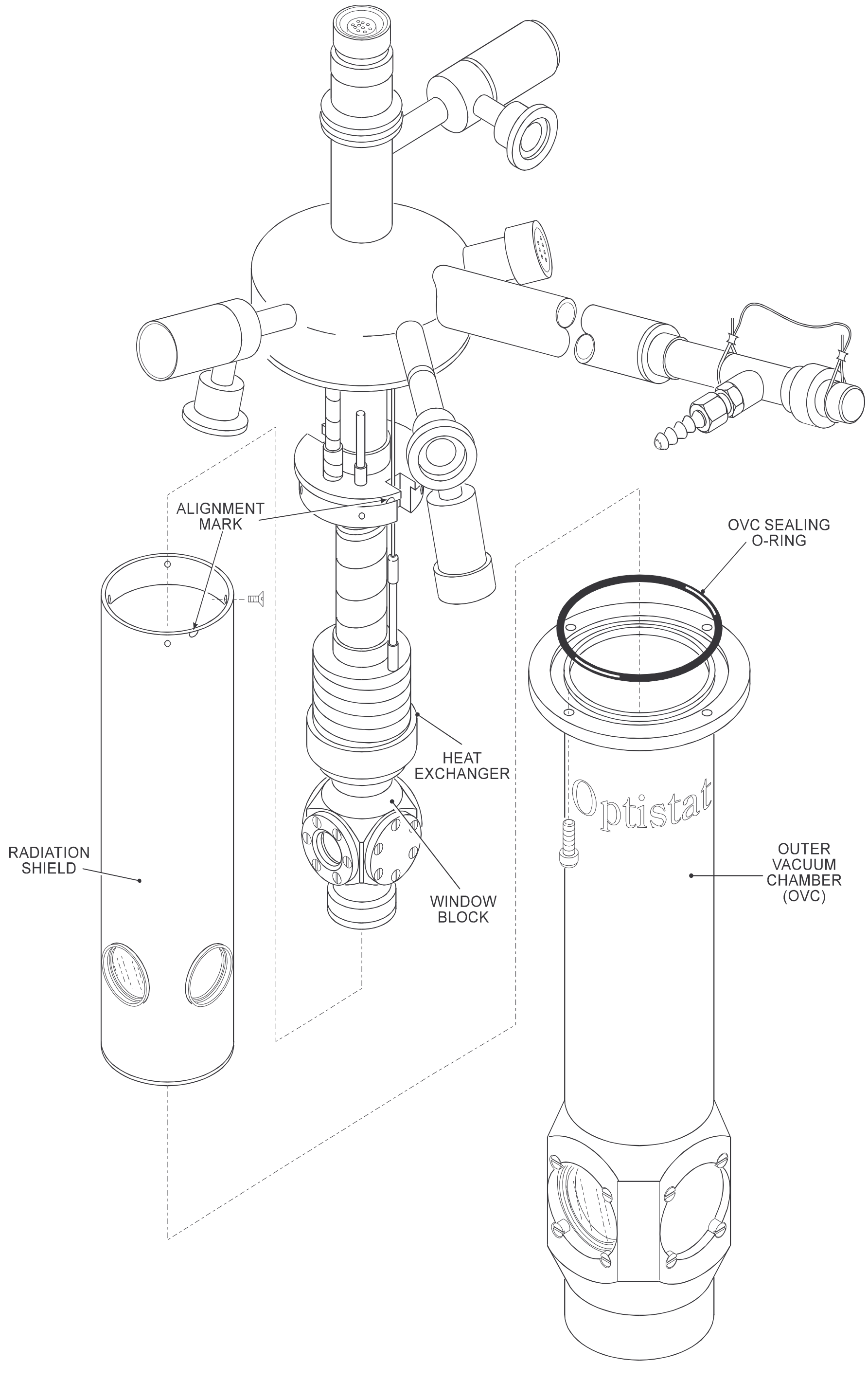

Figure 6 Removing the OVC and radiation shield

6.3 Window replacement

If you need to clean the windows, they can all be removed as follows.

6.3.1 Outer windows

Carefully remove the four nylon window retaining screws. The outer windows are shown in Figure 7. If the cryostat is turned over, the window will fall out. Clean it with a suitable lens cleaner and lens tissue. Before the window is replaced, check the 'O' ring, clean and grease it lightly with vacuum grease. The windows should be carefully tested for leaks with a mass spectrometer leak detector if one is available. However, some window materials (for example polythene, mylar, and aluminised mylar) are porous to helium gas at room temperature, and cannot be leak tested in this way.

6.3.2 Outer windows with retaining rings

The outer windows of this type of cryostat normally rely mainly on the vacuum inside the cryostat to keep them in place. The four nylon screws are there to prevent the windows falling out, but are not designed to press hard enough to secure the windows against the sealing 'O' ring. This approach ensures that no unnecessary strain is induced in the windows.

In certain circumstances, however, the outer vacuum chamber itself is required to fit inside an evacuated environment. In this case the nylon screws are replaced by stainless steel screws, and a plastic retaining ring prevents the screws from damaging the window.

To secure the window in these cases, fit the retaining ring over the window, and secure it with six M3 stainless steel screws and stainless steel washers. The screws should be tightened evenly so that the window is held against the 'O' ring, but in no circumstances should they be tightened further as this could damage the window.

6.3.3 Radiation shield windows

The radiation shield windows are held in place by wire clips as shown in Figure 8. Carefully prise the clip out of the window frame, and turn the cryostat over to let the window drop out. When you replace the window it should not be loose in the mount. The pressure from the wire clip should be sufficient to make sure that the window is cooled properly.

6.3.4 Indium sealed inner windows

Indium sealed windows are suitable for operation at temperatures up to 320 K. They may be removed and replaced as follows.

Vent the OVC and remove the OVC and radiation shield. Vent the sample space. Undo the retaining screws carefully. Remove the window by carefully prising the window frame away from the sample block to break the seal. Remove the old indium from the sealing surfaces using a soft scraper (for example a small piece of wood). Do not scratch these surfaces, as this would probably cause a leak.

Clean the windows with an appropriate lens cleaner.

Clean a piece of indium wire, cut it to the required length, and lay it in the indium groove as shown in Figure 9. The wire should overlap by about 3 mm.

Place the window and frame on the sample block, and tighten the screws evenly until the faces are in close contact. Tighten each screw and then the one opposite, so that the frame is not distorted.

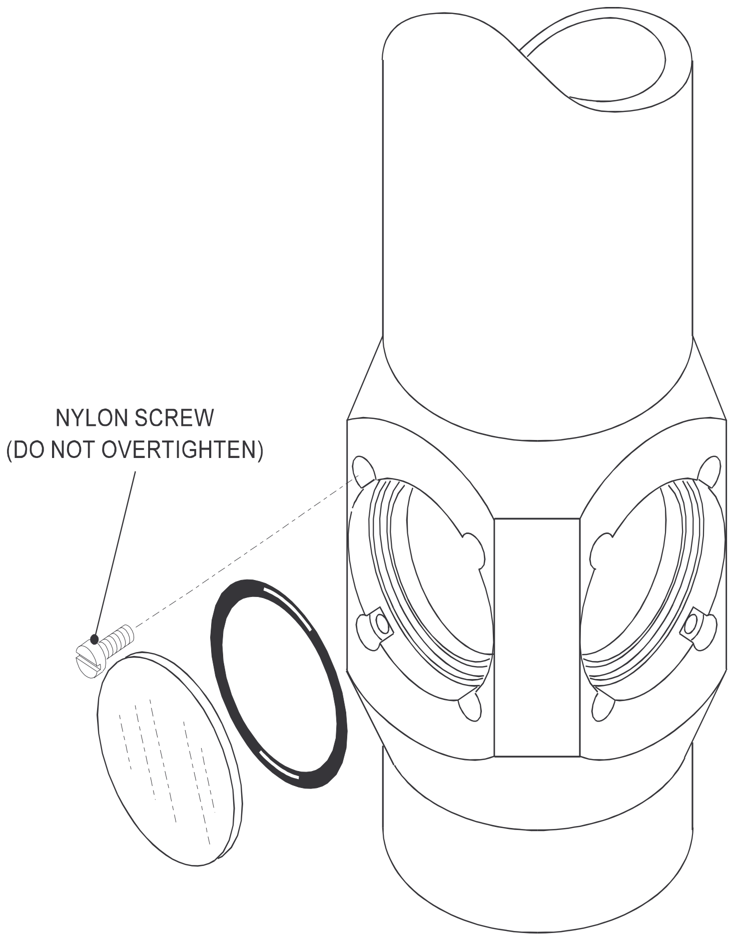

Figure 7 'O' ring sealed outer windows

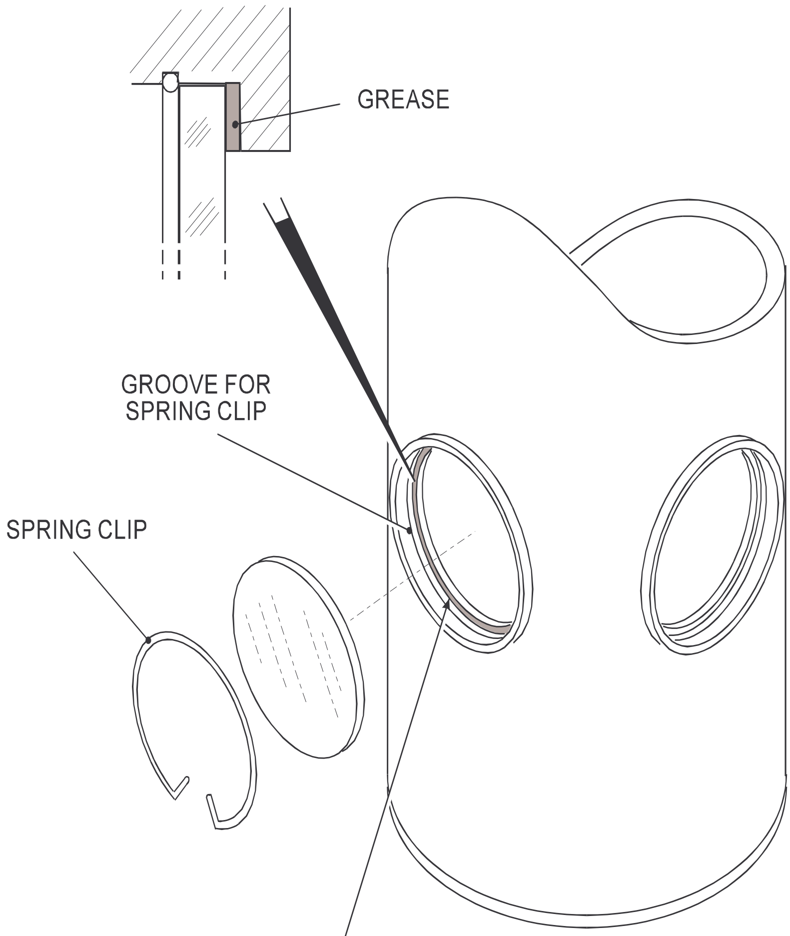

Figure 8 Radiation shield windows

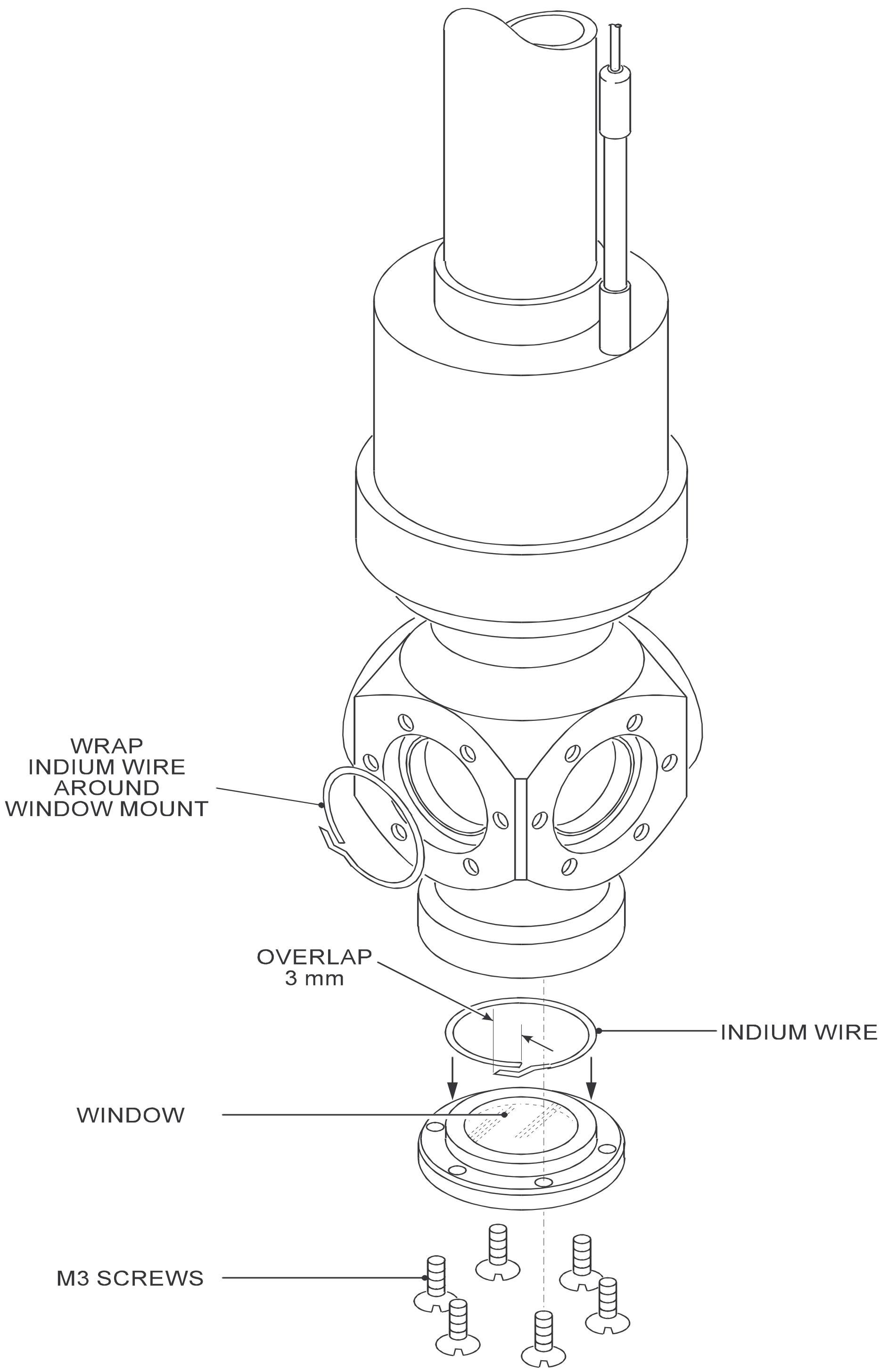

Figure 9 Indium sealed inner windows

6.3.5 Copper gasket sealed inner windows

Copper gasket sealed windows (or metal window blanks) may be used at temperatures up to 500 K. They may be removed and replaced as follows.

Remove the windows as described in section 6.3.4

Clean a new copper gasket thoroughly. The spare gaskets supplied by Oxford Instruments are thoroughly annealed, and if you make your own gaskets you should remember to do this. Put the gasket in the recess on the sample block, and position the window and frame over it. If possible, use new high tensile steel screws, and tighten them down evenly. Tighten each screw and then the one opposite, so that the frame is not distorted.

7 Electrical connections on the cryostat

The standard cryostat is fitted with a ten pin seal on the top plate. This is used for the connection to the heat exchanger. The seal is held in place by the black knurled nut. Do not remove it unless you need to gain access to the wiring.

7.1 Standard wiring for an OptistatCF with a rhodium iron resistor

As standard, the heat exchanger is fitted with a rhodium iron resistor (set up for four wire measurement) and two heaters. Detailed information about the thermometer is given at the end of this manual.

Pin	Function
A	) Heat exchanger heater
B	) (Watlow Firerod heater)
C	) V+
D	) V- Heat exchanger sensor
E	) I+
F	) I-
H	)
J	) Spare
K	)
L	)

7.1.1 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	Expected resistance
A to B	15 - 25 Ω
C to D	30 to 40 Ω
C to E	< 15 Ω
C to F	30 to 40 Ω
E to F	30 to 40 Ω
A to C	> 1 MΩ
A to ground	> 1 MΩ
C to ground	> 1 MΩ

7.2 Wiring for an OptistatCF with a thermocouple

OptistatCF cryostats are available with special wiring, and the most common option is to replace the rhodium iron resistor with a AuFe (0.07%)/Chromel thermocouple. A special thermocouple feedthrough is used so that the thermocouple wire is continuous. Two leads emerge from the hood which covers the feedthrough, as shown in Figure 10.

- The reference junction, approximately 1 m long, covered in a coloured sleeve
- The temperature controller lead, approximately 10 cm long.

The reference junction should be held at a known temperature. It should normally be put into liquid nitrogen (at 77 K) while the cryostat is running. The other lead is fitted with a ten pin connector to suit a standard Oxford Instruments temperature controller lead.

Pin	Function
A	) Heat exchanger heater (Watlow Firerod)
B	)
C	) Thermocouple output
D	)

If you suspect that one of the thermocouple wires is broken you may find it difficult to repair it, and prefer to have it repaired by one of our service engineers.

Caution: If the reference junction is accidentally allowed to warm above 77 K the temperature controller will increase the power supplied to the heater, and it may warm the cryostat above its maximum safe working temperature. A safety cut out switch is supplied but it is not good practice to rely on it.

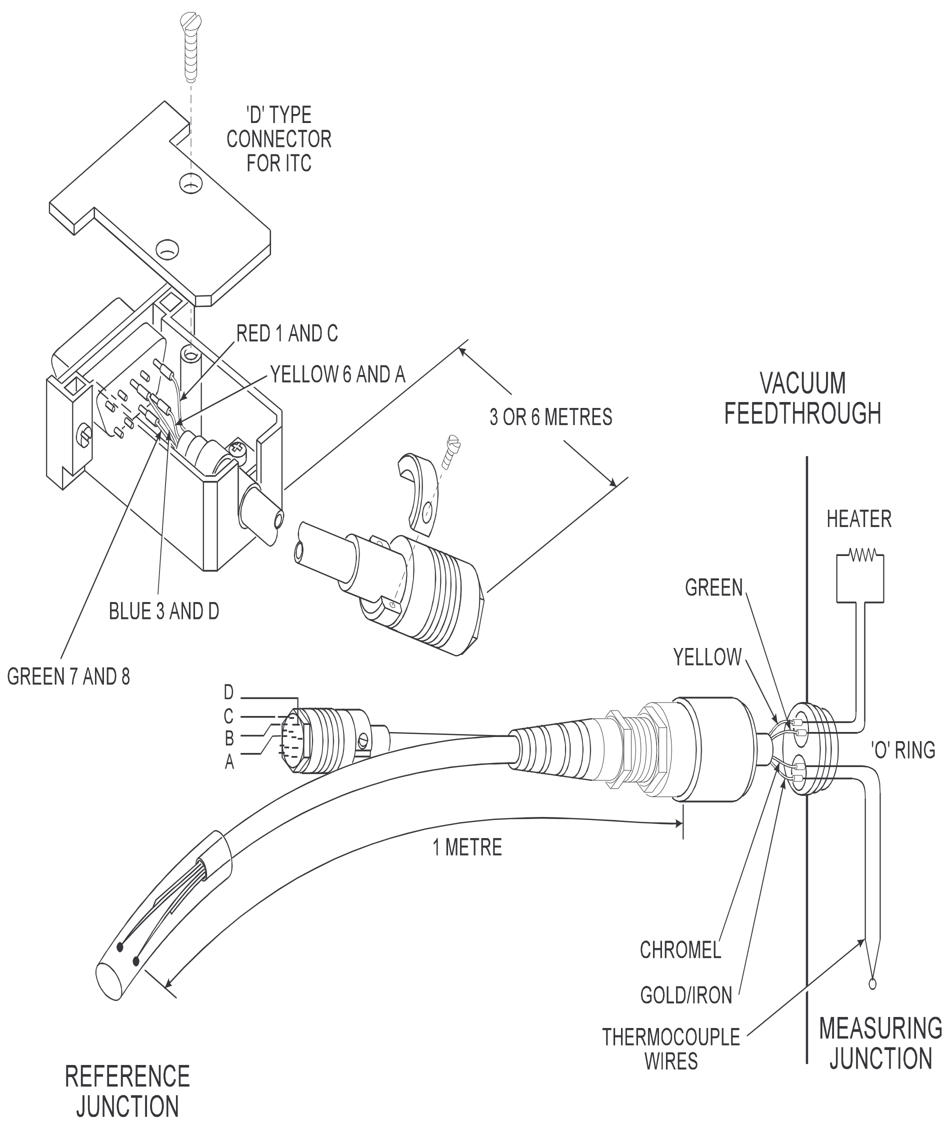

Figure 10 Electrical connections to an OptistatCF with a thermocouple

8 Fault finding

The following table shows the most common faults on the OptistatCF cryostat. Refer to the fault finding table in the transfer tube manual.

Symptom	**Diagnosis and suggestions**
Cryostat OVC cannot be pumped to high vacuum Or Water condenses on the cryostat body when it is cold	Check the cryostat OVC for leaks. In particular check: • Cryostat top plate seals • Outer window seals Also check the inner windows by pumping the sample space. If the pressure in the OVC then drops, check the inner windows individually and replace the gaskets as appropriate. 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.
Sample space cannot be pumped to high vacuum	Check for air leaks around the fittings at the top of the sample space.
Low flow rate through the transfer tube	There may be a blockage in the flow path. Loosen the transfer tube nut and watch the flow gauge to see whether the flow increases. If it does not, the transfer tube is likely to be blocked. Warm the blocked part of the system to room temperature and blow warm dry helium gas through it to remove moisture.
Cryostat will not cool down	Check whether there is any flow of gas through the system, using the gauge on the VC31 if applicable. If not see above.

Symptom	Diagnosis and suggestions
Cryostat will not reach base temperature	Check that the heater is switched off. 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. Check the connections to the thermometer and make sure that it is working properly and in good thermal contact with the cryogen flow. Check that you have not added too much heavy wiring to the sample holder, introducing a high heat load. Check the quality of the vacuum in the OVC Check the vacuum in the transfer tube Check the cryostat for mechanical damage. Warm it to room temperature and remove the OVC to check whether the radiation shield touches the sample space or OVC.
Air or moisture condenses on the inner windows	Check whether: • The sample space valve is shut • The sample space 'O' ring has been damaged Or Did you allow some air into the sample space when you changed the sample?
Sample rod cannot be removed when the cryostat is cold.	Air may have been condensed into the sample space, freezing the sample holder in place. Connect a pump to the sample space valve, and pump the sample space while you slowly warm the cryostat to room temperature. Check the sample space for leaks before you run the system again.
Cryostat cannot be warmed up from base temperature. or Heater not working	Check that the 'set temperature' is higher than the present sample temperature, or switch the heater on manually. Check that the heater voltage limit on the temperature controller is high enough. 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.
Sensor not reading correctly	Check the wiring

8.1 Additional fault finding for version with low temperature entry arm

Symptom	Diagnosis and suggestions
No flow or low flow rate through the rotary pump	Check that the needle valve on the cryostat arm is open far enough. If you still cannot increase the flow sufficiently, it is likely that the narrow tube to the heat exchanger is blocked, and you will have to warm up the cryostat and purge it with clean dry helium or nitrogen gas to remove the contamination.