

*Variox<sup>BL</sup> variable temperature cryostat  
(Static versions)*

*Operator's Handbook*

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### ***Warnings***

Before you attempt to install or operate this equipment for the first time, please make sure that you are aware of the precautions that you must take to ensure your own safety.

### ***Important 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.

## **1. Introduction**

### **1.1. Introduction to this manual**

Variox<sup>BL</sup> is a bath cryostat with a variable temperature facility built in. A range of tail sets is available for different experimental requirements. The sample is loaded through the top of the cryostat on a sample rod and is cooled through exchange gas in the sample space (static system). The sample temperature is continuously variable between 1.5K and 300K or 1.5K and 400K, depending on customer's requirement.

This manual is designed to describe the static Variox<sup>BL</sup> with either optical or non-optical (plain) tail sets. It explains how to operate it with an *Oxford Instruments* ITC502 or ITC503 temperature controller. You can run the system manually if a temperature controller is not available but it is difficult to obtain good temperature control.

We recommend that you read this manual carefully before you try to operate the system. With practice you will find the best way to use the system for your own special application.

### **1.2. Safety requirements**

Please refer to the separate booklet *Safety Matters* supplied with the system. This includes information about the properties of liquid nitrogen and liquid helium, and detailed recommendations about the precautions you should take. It is your responsibility to ensure your own safety and the safety of the people working around you.

## **2. Description of the system**

### **2.1. Introduction to static Variox<sup>BL</sup> systems**

The Variox cryostat contains a liquid helium reservoir, a liquid nitrogen reservoir and a variable temperature insert. The sample is loaded into a central tube which can be filled with exchange gas (usually helium to give good thermal contact). Liquid helium is supplied from the reservoir to the heat exchanger through a needle valve, allowing the flow to be optimised to suit the operating requirements. The helium then passes through an annular space around the sample space and out through the exhaust port at the top of the insert.

The helium reservoir has three necks, used for different services. One is used for the needle valve drive rod; another accepts a 3/8" (9.6mm) diameter liquid helium transfer tube, used to fill the helium reservoir; the third is designed to accept a 3/16" (4.8mm) diameter helium level probe. The exhaust ports of the helium reservoir are connected to a manifold at the top of the cryostat. A pressure relief valve is fitted to this manifold to release helium gas to prevent a dangerously high-pressure building up in the reservoir.

A liquid nitrogen reservoir is used to cool a radiation shield around the liquid helium reservoir and sample space. This shields the low temperature parts of the system from room temperature thermal radiation. It also shields the variable temperature insert from the liquid helium reservoir so that the helium evaporation rate is not affected much by high sample temperatures.

The nitrogen reservoir has three vents. One of these must be fitted with a non-return valve whenever the system is cold. This ensures that evaporating nitrogen gas can be released safely from the reservoir even if the other necks are accidentally blocked by ice condensed from the atmosphere. The non-return valve is sometimes built into a liquid nitrogen level probe.

The variable temperature insert is built into the cryostat. It uses a continuous flow of liquid helium from the reservoir to provide cooling power. A heat exchanger at the bottom of the sample space is fitted with a heater and thermometer, so that the temperature of the helium flowing through the insert can be controlled by a temperature controller. This helium then flows out of the cryostat to a small vacuum pump, which is used to promote the flow.

If the cryostat is fitted with an auto needle valve the ITC temperature controller can be used to optimise the helium flow rate automatically over most of the operating temperature range. This ensures that the system is running as economically as possible. The top plate is fitted with other services including:

- electrical access to the heat exchanger thermometry and needle valve heater
- a vacuum valve for the outer vacuum chamber (OVC)
- pressure relief valve to protect the OVC (either built into the above vacuum valve or welded onto the top plate).

## **2.2. Tail sets**

A range of optical and plain tails is available to suit different experimental requirements. These tail sets consist of the variable temperature insert, a nitrogen cooled radiation shield and an outer vacuum chamber tail. The insert tail has an isothermal region around the sample position, supported on a thin walled stainless steel tube.

### **2.2.1. Plain tails (no optical access)**

The outside diameter of the tail has been made as small as possible, consistent with the required sample space diameter.

A Firerod heater and **Cernox** sensor are fitted to the sample space heat exchanger as standard on plain tailed cryostats.

### **2.2.2. Optical tail set**

These tails give radial optical access to the sample space. You can choose from a range of window materials to suit your experimental application.

The inner and OVC windows are glued and the radiation shield windows are fixed by tape.

A Firerod heater and a **Cernox** sensor are fitted to the sample space heat exchanger as standard on optical tailed systems.

## **2.3. Sample Access**

The sample is mounted on a sample rod (if supplied) and loaded into the cryostat through an NW50 Klein flange. The sample can be rotated about the cryostat axis by slackening the clamp, but care must be taken not to lift the sample rod and break the seal while the system is cold.

### ***3. Unpacking and assembling the system***

#### ***3.1. Unpacking***

The system is delivered in a strong wooden crate standing on a transit pallet. Handle it carefully when you move it into your laboratory.

**Note**      **Packing is also fitted inside the cryostat to support it in transport.**

**Caution**                      **Do not stand the cryostat on its tail. The tail has a thin section which is not capable of supporting the weight of the system.**

Remove the crate top panel and then the front panel. Remove the two wooden split ring support pieces and carefully slide the cryostat out. Clear a suitable horizontal working surface and lay the cryostat horizontally on the surface making sure that it cannot accidentally roll.

Inspect the system for any signs of damage and consult your local agent or *Oxford Instruments* directly if any are observed.

**Caution**      The main helium bath exhaust pressure relief valve may have been supplied separately. It must be fitted before the system is used.

If possible, keep the packing in case the system should need to be shipped in the future.

#### ***3.2. Removing internal transit packing***

The cryostat has been fitted with internal packing to prevent damage while the cryostat is being transported. All packing that has to be removed is painted red. A diagram on the outside of the cryostat indicates where to find the packing. Any other spacers should be replaced if you have to remove them.

Lay the cryostat horizontally as described above. The cryostat is usually shipped under vacuum, so you have to allow it up to atmospheric pressure. Slowly open the outer vacuum chamber (OVC) valve to allow air into the OVC.

Use one of the 'Allen keys' (from the spares kit) to remove the cap-head screws counter bored into the bottom of the OVC base flange. Carefully support the tail when you remove the last screws so that it does not drop onto the radiation shield tail. Carefully remove the OVC tail. The radiation shield is now visible. Inspect it and its windows (if fitted) for signs of damage.

**Tip**      Notice the alignment marks on the mating flanges, so that the tails can be re-aligned properly during assembly.

The radiation shield is fixed to the liquid nitrogen reservoir by SS cap-heads screws. It is highly polished to optimise the thermal performance of the cryostat. Always wear gloves while you are handling internal parts of the cryostat so that they are kept clean.

Remove three packing disc from radiation shield. Remove the radiation shield by removing the screws. You should now be able to see the red packing between the helium reservoir and the nitrogen reservoir. Remove it and keep it for future use. Cover the slots in copper flange using Aluminium tap.

**Caution**      **Be careful as this region contains delicate wiring.**

### **3.3.      *Electrical connections***

The heater and thermometer on the heat exchanger are wired to an electrical connector on the top of the cryostat. A heater on the needle valve is also wired to this connector. This heater is used if the valve becomes blocked with solid air or ice while the system is cold.

Use a suitable digital multi-meter (or equivalent):

- a) to check the sensor continuity
- b) to check the heater continuity
- c) to check for a short circuit between sensor and heater
- d) to check for a short circuit between the heaters or sensors and the cryostat body.

If you find any problems during the electrical check , consult your local agent or contact *Oxford Instruments* for advice. Remember that moisture on your fingers can affect the readings if you touch the terminals during these electrical checks. The values given are typical and will vary slightly from one system to another.

#### **3.3.1.      *Optical system or Plain tailed system***

All the cryostat connections are made to a single hermetically sealed 10 pin plug (Figure 1) on the top plate. The pin connections are as follows.

- A     )           Control Heater
- B     )           (Firerod cartridge)
  
- C     )   V+
- D     )   V-    Cernox Sensor
- E     )   I+
- F     )   I-
  
- K     )           Needle Valve Heater
- L     )



## Wiring Check

Pins	Expected resistance	Test For
A- B	20 $\Omega$	Heater
C - D	37 $\Omega$	V+ V-
C - E	8 $\Omega$	V+ I-
C - F	37 $\Omega$	I+ I-
A - C	>1M $\Omega$	Heater-Sensor
A - Ground	>1M $\Omega$	Heater-Cryostat
C - Ground	>1M $\Omega$	Sensor-Cryostat
K - L	70 $\Omega$	Needle valve heater (where present)

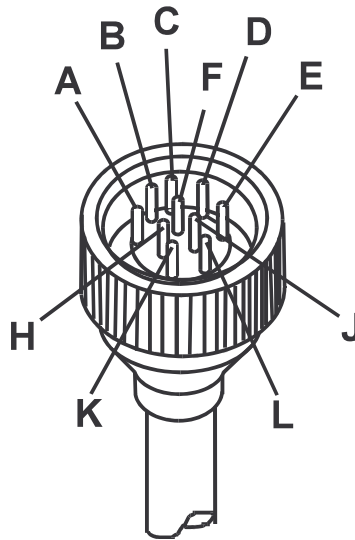


Figure 1 Ten pin seal

### 3.4. *Re-assembling the tails*

Before you start to re-assemble the tails, locate the alignment marks on the tail flanges and the cryostat. Replace the radiation shield tail, ensuring that the screws are tight. Polish or clean the shield if the surface is contaminated.

Ensure that the 'O' ring and the groove in the OVC tail are clean and dry. Lightly grease the 'O' ring with vacuum grease. Fit the OVC tail and tighten the cap-head screws uniformly so that there is no uneven force on the 'O' ring as it compresses. It is not necessary to use extreme force on these screws.

## **4. *Preparing the system for operation***

### **4.1. *Evacuating the outer vacuum chamber (OVC)***

The OVC has to be pumped to high vacuum. 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 cryostat 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.

#### **Caution**

Pumping lines used for the OVC of the cryostat or transfer tube should not be used for pumping helium gas. It is difficult to remove the helium thoroughly, it may contaminate the high vacuum chambers and adversely affect their insulation performance.

### **4.2. *Evacuating the transfer tube (if supplied)***

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.

### **4.3. *The 3-way valve***

The sample space is fitted with a 3-way valve to control the flow of gas into or out of the sample space. The knob can be moved to three positions:

- pointing horizontal on right, the sample space is connected to the vent on top of the valve
- upwards, the sample space is closed off
- pointing horizontal on left, the sample space is connected to the helium reservoir exhaust

You can connect a pump to the vent port and use it to pump gas out of sample space.

### **4.4. *Rotary pump connections***

Connect a rotary pump to the sample space heat exchanger pumping port (NW25 flange) as shown on Figure 2. You may also like to be able to connect a supply of helium gas into this line.

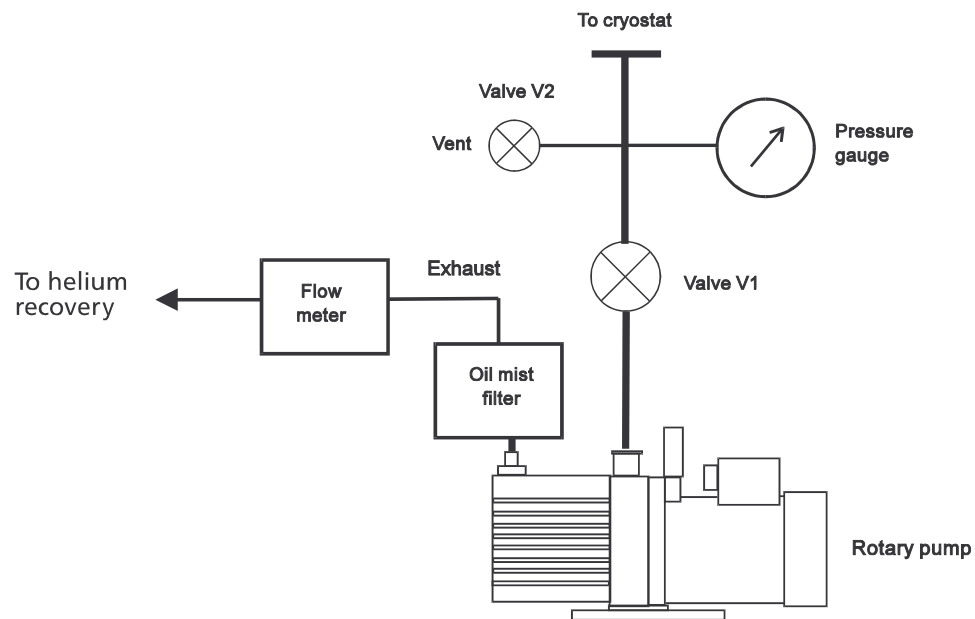


Figure 2 Pumping system for the Variox cryostat

If valve V1 is a diaphragm valve it should be connected close to the pump as shown on the diagram. This allows the gas to warm to room temperature before it passes through the valve. If it is fitted close to the cryostat it might be damaged by the cold gas.

The exhaust from the rotary pump can be connected to a 0 – 500 cm<sup>3</sup>/min helium gas flow gauge (0 – 0.5l/hr liquid helium equivalent) with suitable filter but should not be connected until base temperature has been reached. The helium gas from the cryostat can be collected in a suitable 'recovery system' if you have one.

#### ***4.5. Pumping and flushing***

Before you cool down the system it is important to remove air from the helium reservoir and heat exchanger so that it cannot freeze and block the narrow capillary tubes. Pump the main reservoir and the heat exchanger to a rough vacuum and then fill them to atmospheric pressure with helium gas (either from a recovery system or from a bladder connected to the exhaust port of the main reservoir). Repeat this process. Pump the sample space to a rough vacuum and fill it to atmospheric pressure with helium gas, by attaching a bladder full of helium gas to the pump out port.

#### ***4.6. Cooling the cryostat***

##### ***4.6.1. Precooling with liquid nitrogen***

The following equipment is required:

- a nitrogen delivery tube (supplied with the system)
- a suitable tube to transfer liquid nitrogen
- a liquid nitrogen storage vessel

Push the liquid nitrogen delivery tube through the transfer tube entry port until it touches the bottom of the main reservoir. Raise it slightly and tighten the knurled nut on the entry port to hold it in place. Connect the delivery tube to the liquid nitrogen storage vessel. It is safest to use flexible stainless steel tube.

**Warning**      **Avoid using rubber or polythene tubes. When these materials are cooled to 77K they become very brittle and can shatter easily.**

Leave the needle valve open. This will reduce the chance of the needle valve becoming jammed and make sure that the valve on the pump (V1) is closed. This will prevent the liquid flowing from the main reservoir into the heat exchanger. Open the exhaust of the liquid helium reservoir to air and transfer liquid nitrogen into the cryostat until it is full. Liquid will overflow through the main reservoir exhaust port. Stop the transfer and leave the system to cool thoroughly for at least one hour. If an excessive amount of ice collects on the manifold you can use a hot air blower to melt the ice; but do not overheat it.

After about one hour, connect the nitrogen delivery tube in the helium reservoir to one of the liquid nitrogen reservoir ports using a suitable tube.

Close the needle valve and pressurise the helium reservoir with helium gas through the main bath exhaust port. 100 to 200 mbar (above atmospheric pressure) should be sufficient to transfer the liquid nitrogen.

If there is still liquid in the helium reservoir when the nitrogen reservoir is full this can be returned to a storage dewar.

When the helium reservoir is empty, pump and flush it with helium gas. Then pump the heat exchanger to a rough vacuum and open the needle valve to fill the sample space with helium gas. This checks that the needle valve and capillary tubes are not blocked.

#### **4.6.2. *The helium transfer***

Ensure that the transfer tube vacuum is satisfactory by pumping it to diffusion pump pressure for several hours.

You will also need a storage vessel of liquid helium and a dipstick/level probe. You should also be able to pressurise the storage dewar. A soccer ball bladder can be used for small dewars; a helium gas cylinder is needed for large storage dewars.

If you have a helium recovery system connect the helium reservoir exhaust manifold to it. If there is no recovery system, ensure the main reservoir is free to vent to air, but air must not be allowed to enter the reservoir freely. If a helium level meter and probe have been supplied, insert the probe into the entry port.

**Warning**      **Ensure that at least one nitrogen port is fitted with a non-return valve.**

**Tip**            Open the needle valve before you start to transfer liquid helium into the system.

Remove the plugs from the system's transfer tube entry port and the top of the storage vessel. Open the exhaust port of the storage dewar to release the pressure as the transfer tube leg is cooled down. Insert the transfer tube legs into the system and into the storage dewar slowly, allowing the dewar leg to cool gradually. Make sure that the end of the transfer tube in the cryostat reaches the bottom of the helium reservoir.

Close the exhaust line on the storage dewar and pressurise it slightly to start the liquid transfer. (This is generally done by gently squeezing a rubber bladder attached to the exhaust of the helium storage vessel). The transfer rate should be such that the exhaust pipe is frozen for not more than 1.5 m (6 ft.) of its length. The initial transfer rate should be equivalent to between 4 and 10 litres of liquid per hour.

When liquid starts to collect in the helium reservoir the exhaust gas flow rate will be seen to drop noticeably (as the ice on the recovery line starts to melt). This typically happens after 15 or 20 minutes. The pressure on the storage dewar can then be increased to transfer the liquid more quickly.

**Tip**            If you see liquid air dripping from the helium reservoir exhaust line at any time during the helium transfer, this indicates that the liquid is being transferred much too quickly.

Use a helium "dipstick" or a level probe to determine the rising helium level. If a helium level meter and probe have been supplied, the meter will have been set up before it left the factory.

The helium transfer should be stopped when:

- a) The level meter (if supplied) indicates 100%.
- b) The dipstick indicates a level of 38cm (for a 4 litre capacity cryostat only)
- c) There is sudden excessive cold gas exhaust from the reservoir.

Stop the transfer by releasing the pressure in the storage vessel. Remove the transfer tube and replace the bungs.

The booklet 'Elementary Practical Cryogenics' (available from *Oxford Instruments*) contains a list of solutions to the problems commonly encountered in liquid helium transfers. Refer to this booklet if you are having problems.

Remove the dipstick (if you have been using one) and replace the black plug. The cryostat is ready for operation.

If you do not have a helium recovery system fit a non-return valve to the exhaust port of the main reservoir to prevent air from entering it. Check that the nitrogen exhaust ports are free by inserting a warm copper rod.

**Note**      **The area around the needle valve handle (or motor on systems fitted with auto needle valves) is sometimes covered with ice after a long helium transfer. Warm it gently before you try to open the needle valve.**

## **5. *Running the system***

### **5.1. *Cooling the sample***

Connect the temperature controller to the electrical connector on the cryostat using the lead supplied. Connect the pumping lines as shown in Figure 1 on page 11. Close valve V2 and the needle valve, switch on the pump and open valve V1.

Open the cryostat needle valve (either by turning it manually or by using the gas flow on the ITC if an auto needle valve is fitted). You should see a flow of gas from the exhaust of the rotary pump, and the temperature of the sample space should start to drop. Adjust the flow rate to get a suitable cooling rate. Note that it is more economical to cool the sample space slowly (typically 2 to 3 K per minute).

You can use a flow meter on the exhaust of the pump to observe the flow rate through the heat exchanger. Suitable flow rates are shown in the test results for your system attached to the back of this manual. When the sample space reaches approximately 20 K close the sample space valve to isolate it from the supply of helium gas. This prevents helium from condensing into the sample space at lower temperatures. Remember to open it again when you increase the sample temperature to above 20 K again.

It typically takes 60 minutes to cool the sample space to 10 K or below. If you want to check the calibration of the temperature controller you can do this as follows. Cool the system to below 4.2K and allow liquid to collect the heat exchanger. Close valve V1 and open V2 to fill the sample space heat exchanger to atmospheric pressure. Then allow the heat exchanger to vent to air freely, so that it reaches its normal boiling point. Check that the temperature reading is consistent with the boiling point of liquid helium at atmospheric pressure.

### **5.2. *Temperature control above base temperature***

#### **5.2.1. *Introduction***

You can control the temperature of the sample space heat exchanger (and the sample) between base temperature and 300 K.

**Note** **When you are operating the system at temperatures below 20 K you should isolate the sample space from the supply of helium gas. This ensures that liquid helium does not condense in the sample space. At higher temperatures the valve should be opened again to allow the gas to expand freely.**

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 procedure for optimising the PID values and control theory are given in the temperature controller manual.

#### **5.2.2. Controlling at a 'set temperature'**

Switch on the pump and ensure that the valve between the pump and the heat exchanger are open. Check that the cryostat has been connected to the temperature controller. Select the channel on the temperature controller corresponding to the sensor which will be used to control the system. 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 base temperature 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 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 needle valve, 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:

- less than 2 volts when the system is working near base temperature
- less than 8 volts when it is working below 300K
- greater than 8 volts when it is working above 300K

#### **5.2.3. Operation above room temperature**

Before you set a temperature above room temperature, check that your system is suitable for this temperature range. The windows and sealing method restrict the upper working temperature on some optical Variox systems to 320K.

You can now control the system at a 'set temperature' as described in section 5.2.2. You may find that you can close the needle valve fully if you are controlling at a high temperature.

### **5.3. Temperature control below 4.2 K**

If you use a diaphragm pump (for example an *Oxford Instruments* GF3 pump a minimum temperature of 3.7 K can be achieved. A larger displacement pump (typically 25m<sup>3</sup>/hr) is required if you want to reach lower temperatures. The Variox system will typically reach a base temperature of 1.4 K in 'single shot' mode or 1.6 K continuously if a large enough pump is used, with suitable pumping lines.



Operation below 4.2 K can be accomplished in two ways.

**5.3.1. *The continuous flow method***

If the sample space is at 4.2 K or above you can set the temperature to control below 4.2 K as follows. Use the needle valve manually to increase the flow rate until the sample space temperature is slightly below the temperature that you want to set. Then use the temperature controller to set the required temperature. Leave the gas flow in manual mode but put the heater into AUTO mode.

The base temperature in continuous flow mode is slightly higher than the base temperature that can be reached in 'single shot' mode, as described below.

**5.3.2. *The single shot method***

Allow the heat exchanger to fill with liquid helium by fully opening the needle valve for a few minutes. You can partly close valve V1 if necessary to allow the pressure to rise in the sample space. However, it is best to start the single shot from the lowest possible temperature if you want to maximise the hold time.

Close the needle valve and adjust the pumping speed to vary the temperature at the heat exchanger. If valve V1 is fully open you should reach the base temperature of the system (for your configuration of pump and pumping lines). Base temperature will be maintained until all the liquid helium in the sample space has evaporated.

The hold time at base temperature may not be very long on static systems.

**5.4. *Changing samples***

The sample is mounted on a sample rod which is used to load the sample into the cryostat and support it in the correct position. The sample rod can be removed and replaced while the cryostat is at any temperature. However, remember that your sample may be subjected to thermal shocks if it is warmed or cooled too quickly. It will also be covered with ice if it is very cold when it is removed from the sample space.

Switch off the pump or close valve V1, open valve V2 or the needle valve and allow the heat exchanger pressure to reach 1 atmosphere. Make sure that the sample space is at atmospheric pressure. The best way to do this is to check that the bladder attached to the sample space valve is partially inflated and that the valve is open. Remove the Klein flange clamp from the top of the sample space.

When the sample space is opened, air and moisture may be allowed in, and these will contaminate the sample space and may fog the windows. The best way to prevent this is to connect a supply 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. A bladder of helium gas attached to the sample space valve will usually be sufficient.

Seal the sample space as soon as possible, using either a sample rod or a blank flange. Follow the same procedure again to put the sample rod back into the sample space.

### **5.5.     *Clearing a needle valve blockage***

A heater is mounted on the needle valve body in case the needle valve becomes blocked with solid air or ice during operation. It is often possible to clear the blockage without warming the whole system to room temperature. Power can be supplied to the heater by the temperature controller if you connect the orange lead in series with the standard controller lead. Pump the heat exchanger and watch the flow meter. Energise the heater gradually until the blockage clears. You should then see the flow through the flow meter increase.

**Caution**     The heater voltage must not exceed 15 volts and should be left there for no longer than 5 minutes. If this does not clear the blockage the system should be warmed up.

## **6. *Warming up the system***

The easiest way to warm up the system is to ensure that the cryogen reservoirs and sample space are free to vent and not open to condensation from the atmosphere and to leave the system to warm naturally. If the cryogen is not re-filled the system will reach room temperature after a few days.

If you have to warm up the system more quickly than this transfer all the cryogen out of the system. The transfer tube should be inserted in the entry port and pushed to a few millimetres above the bottom of the reservoir. Pressurise the reservoir slightly to blow the liquid out of the system.

When the liquid has all been blown out of the system, remove the transfer tube and allow the system to warm up naturally. Leave the needle valve open and leave the heat exchanger exhaust closed off.

## **7. Maintenance**

### **7.1. Rubber 'O' Rings**

Whenever you remove part of the outer vacuum chamber, inspect the 'O' rings for signs of damage. A damaged 'O' ring should be replaced.

If you suspect a leak, check any 'O' rings that have been disturbed. There is a moving 'O' ring in the cryostat evacuation valve.

### **7.2. Removal and re-assembly of the OVC and radiation shield tail**

The tail will only need removing to:

- remove or replace the internal packing
- change or clean the windows
- modify or repair the wiring
- repair mechanical damage

The process is outlined in section 3.2.

The procedure for re-assembly of the tails is outlined in section 3.4.

## 8. Possible problems and solutions

SYMPTOM	LIKELY CAUSES	SOLUTIONS
Poor vacuum in OVC	Leak on pumping system	Shut dewar OVC valve and check pumping system base vacuum.
	Leak on dewar or tails	Obtain mass spectrometer and identify source of leak. Check all 'O' rings for cleanliness. Check condition of mylar windows.
	Excessive moisture in OVC	Pump and re-flush with dry nitrogen several times, then pump again.
Condensation or frost on OVC during cooldown	Poor OVC vacuum	Pump OVC again, check for leaks with detector.
Heat exchanger pressure does not rise when needle valve is opened at nitrogen precooling stage or Needle valve jammed or no cooling at heat exchanger	Blocked needle valve or capillaries	The needle valve carries a small heater. Blockages can sometimes be removed by gently heating the needle valve via the heater. Excessive heat may damage the heater. Maximum heater voltage 15 V.
Condensation or frost on transfer tube.	Poor vacuum	Pump it again
Transfer tube shows ice "spots"	Internal capillary touches outer tube	Repair or replace if losses are unacceptably high.
Liquid helium will not collect in main reservoir.	Poor OVC vacuum (cold)	Pump OVC again and test for leaks
	Liquid nitrogen still present in the helium reservoir Faulty transfer tube	Stop helium transfer and remove remaining nitrogen.  Look for cold spots on the outside of the transfer tube. Stop the helium transfer and check the vacuum in transfer tube.
Sample space cools slowly but helium consumption is high (seen at heat exchanger exhaust).	Radiation shield is touching heat exchanger	Disassemble and check radiation shield position.

SYMPTOM	LIKELY CAUSES	SOLUTIONS
Poor temperature stability on automatic set point control	Incorrect PID settings or excessive helium flow through heat exchanger.	See Test Results for correct PID and flow values
Sample rod is jammed in sample tube	Ice or solid air blockage in sample tube	Evacuate sample tube if possible. Flush repeatedly with warm gas. If unsuccessful, gently warm to 300K, while pumping the sample space and heat exchanger exhaust.

## ***9. Test results and details of thermometers***

The test results and details of the thermometer on the system for your system are shown on the following pages.

## Variable temperature insert test results

Type of insert ..... Static

Sample space diameter ..... 25 mm

Temperature range ..... 1.6 – 300 K

Displacement of pump used for tests ..... 40 m<sup>3</sup>/hour

## Temperature controller

Type of temperature controller ..... ITC503

Serial number of temperature controller ..... 503SS0187

Heater voltage limit ..... 40 V

Auto needle valve limit ..... 99 %

Auto needle valve set up (C1, C2, C3, C4, C5,C6) ..... 1, 199, 50, 9, 11,0

## Thermometer information

Temperature controller channel	ITC range name	Serial number and calibration number	Resistance at 300 K	Position of sensor
1	C260	X43260/497316	70 Ω	Heat Exchanger
2	-	-	-	-
3	-	-	-	-

## Single shot at base temperature

Base pressure	1.8 mbar
Flow (gas at NTP)	1.1 litre/minute
Temperature controller, channel 1	1.4 K
Temperature controller, channel 2	N/A K
Temperature controller, channel 3	N/A K



## Dynamic base temperature

Pressure	1.9 mbar
Flow (gas at NTP)	0.5 litre/minute
Temperature controller, channel 1	1.44 K
Temperature controller, channel 2	N/A K
Temperature controller, channel 3	N/A K
Auto needle valve position	3.6 %

## Temperature Control Tests

Set temperature (K)	1.6	3.0	4.2	6.0	10.0	20.0	50.0	77.0	100	200	300
Sample holder or heat exchanger temperature (K)	-	-	-	-	-	-	-	-	-	-	-
Time set	11:49	11:12	7:55	13:02	14:01	15:26	17:37	7:10	08:10	9:30	11:34
Time achieved	11:59	11:27	8:20	13:40	14:50	16:00	18:05	7:57	08:29	10:37	12:45
Stability (mK)	10	10	10	10	10	40	40	50	50	50	100
Proportional band (%)	50	50	50	50	50	7.5	6	4.2	4.2	4	3
Integral time constant (minutes)	0.4	0.4	0.4	0.4	0.4	0.5	0.5	0.8	1.1	2	2.5
Derivative time constant (minutes)	0	0	0	0	0	0	0	0	0	0	0
Pressure (mbar)	1.6	1.1	0.9	0.8	0.8	0.7	1.1	2.1	2	2.4	0.7
Gas flow (litres/minute at NTP)	0.5	0.6	0.6	0.5	0.5	0.2	0.5	0.9	0.8	1.1	0.2
Heater voltage (V)	0.3	0.3	0.3	0.4	0.4	0.7	3	5.9	6.7	12.6	16.2
Auto needle valve position (%)	3.4	3.1	2.2	2.6	2.2	1.9	2.9	3.7	3.6	4	4.5
Flow of helium gas from helium reservoir (litres/minute at NTP)	0.6	0.5	0.4	0.4	0.3	0.5	0.7	0.6	0.6	1.2	3.5
He4 Level %	44.7	44.8	46.4	42.8	42.4	100	96.4	88.4	87.1	83.3	79.3
N2 jacket B.O. (l/min of gas at NTP)	2.7	2.7	2.5	2.6	2.7	2.8	2.8	2.7	2.8	2.6	2.8
N2 Level %	96.4	97.1	98.5	92.1	88.5	84.1	80.2	48.8	45.4	40.8	37.3

## Dewar

### Specifications (static conditions)

Dewar type	VarioxBL
Liquid helium reservoir evaporation rate at 4.2K	150 cc/hr
Liquid nitrogen reservoir evaporation rate	300 cc/hr .....

### Dewar test results

Useful liquid helium volume		13	Litres
Liquid helium evaporation rate at 4.2K %	46	100	cc/hr
Useful liquid nitrogen volume		10	Litres
Liquid nitrogen evaporation rate at %	98	215	cc/hr

### Weight and dimensions of the system

Approximate weight of the system	90 kg
Minimum ceiling height requirement for sample loading	4.1 m